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1-[(2,5-Dioxopyrrolidin-1-yl)(phenyl)-methyl]urea

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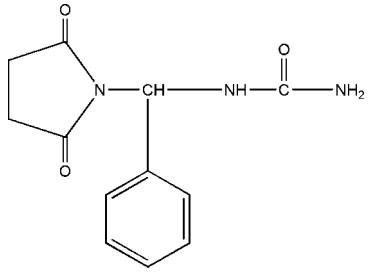
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.089; data-to-parameter ratio = 11.9.

In the title compound, $C_{12}H_{13}N_3O_3$, strong intermolecular N—H···O interactions link the molecules into centrosymmetric dimers. The crystal packing is further stablilized by van der Waals forces.

Related literature

For related literature, see: Tramontini (1973); Tramontini & Angliolini (1990); Wozniak *et al.* (2000).



Experimental

Crystal data

$C_{12}H_{13}N_3O_3$
M_r = 247.25
Monoclinic, $P2_1/n$
a = 13.8460 (4) Å
b = 5.3527 (1) Å
c = 15.9052 (4) Å
β = 101.310 (1)°
V = 1155.90 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.30 × 0.15 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.894, T_{max} = 0.982
11730 measured reflections
2032 independent reflections
1620 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.089
S = 1.04
2032 reflections
171 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.86	2.19	2.9600 (18)	149
N3—H3B···O3^{i}	0.929 (9)	2.489 (13)	3.2867 (19)	144.1 (13)
N3—H3A···O3^{ii}	0.940 (9)	2.037 (9)	2.9655 (17)	169.0 (16)

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+1, -y+2, -z$.

Data collection: *SMART* (Bruker–Nonius, 2004); cell refinement: *SAINT-Plus* (Bruker–Nonius, 2004); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank the Department of Chemistry, IITMadras, Chennai, India, for the single-crystal X-ray diffraction data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2077).

References

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supplementary materials

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1-[(2,5-Dioxopyrrolidin-1-yl)(phenyl)methyl]urea

S. Rajeswari, G. Venkatesa Prabhu, K. Krishnakumar, V. Ramkumar and B. Varghese

Comment

Mannich bases- compounds with the general formula R—CH_2—N< have been studied extensively, because of their applications in pharmaceutical and polymer chemistry. They are very good model systems for both intermolecular and intramolecular hydrogen bonding and a variety of other effects (Wozniak *et al.*, 2000). In the crystal structure of the title compound, the molecules which are related through center of inversion are linked to each other through N3—H3···O3 hydrogen bonds (2.363 (11) Å, 155.1 (15) °). They form a dimeric pair. These dimeric pairs are linked to its b-translation equivalent through N2—H2···O2 (2.19 Å, 149.4 °) hydrogen bonding. The hydrogen bonded dimer and the translation equivalents form an one dimensional dimeric chain running parallel to the *b* axis. The one dimensional chains are linked through van der Waals interactions. Succinimide moiety is almost planar with maximum deviation of 0.032 Å for the N atom from the least squares mean plane of atoms. The planarity can be attributed to the sp^2 hybridization of C1 and C4 atoms.

Experimental

Urea (12 g, 0.2*M*), Succinimide (19.8 g, 0.2*M*) and Benzaldehyde (22 ml, 0.2*M*) were taken in equimolar ratio. A concentrated aqueous solution of urea and succinimide was prepared. Benzaldehyde was added in drops with continuous stirring of the solution. The mixture first became oily and then slowly turned into a white crystalline mass, which was separated by suction filtration and washed several times with water. The product was dried and recrystallized using acetone by slow evaporation (Tramontini, 1973; Tramontini & Angliolini, 1990).

Refinement

All the H atoms except those of N atoms were geometrically fixed at chemically meaningful positions. The hydrogen atoms of the phenyl ring were allowed to ride at a distance of 0.93 Å from the parent carbons and their thermal parameter were fixed at 1.2 times that of the parent atom. The secondary CH_2 hydrogen were fixed at a distance of 0.97 Å from the parent atom and their thermal parameters were fixed at 1.2 times the parent atom. The H atoms associated with nitrogen atoms were located from the difference fourier map and refined. However, their distances were constrained from the parent atom to avoid abnormal geometry.

Figures

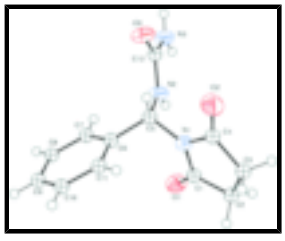

Fig. 1. *ORTEP* representation of the molecule showing the atom numbering scheme. Thermal ellipsoids are drawn with 30% probability.

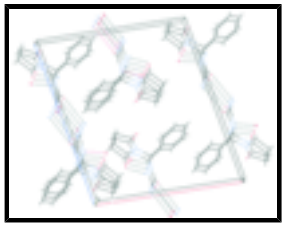

Fig. 2. Packing diagram of title compound projected down the *b* axis

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Crystal data

$C_{12}H_{13}N_3O_3$	F_{000} = 520
M_r = 247.25	D_x = 1.421 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3812 reflections
a = 13.8460 (4) Å	θ = 2.6–26.8º
b = 5.3527 (1) Å	μ = 0.11 mm^{-1}
c = 15.9052 (4) Å	*T* = 298 (2) K
β = 101.310 (1)º	Needle, colourless
V = 1155.90 (5) Å^3	0.30 × 0.15 × 0.10 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2032 independent reflections
Radiation source: fine-focus sealed tube	1620 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.031
T = 298(2) K	θ_{max} = 25.0º
phi and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (Blessing, 1995)	*h* = −16→15
T_{min} = 0.894, T_{max} = 0.982	*k* = −5→6
11730 measured reflections	*l* = −18→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.036	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.089	$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 0.3355P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
2032 reflections	$\Delta\rho_{max}$ = 0.13 e Å^{-3}

171 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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}
C1	0.42128 (11)	0.9642 (3)	0.38640 (9)	0.0335 (4)
C2	0.33912 (12)	0.8629 (3)	0.42524 (11)	0.0460 (4)
H2A	0.2851	0.9812	0.4189	0.055*
H2B	0.3619	0.8282	0.4857	0.055*
C3	0.30687 (12)	0.6246 (3)	0.37642 (11)	0.0422 (4)
H3D	0.3205	0.4804	0.4138	0.051*
H3E	0.2369	0.6291	0.3523	0.051*
C4	0.36532 (11)	0.6135 (3)	0.30708 (10)	0.0352 (4)
C5	0.50746 (11)	0.8393 (3)	0.26626 (9)	0.0324 (4)
H5	0.5056	0.6887	0.2310	0.039*
C6	0.61091 (11)	0.8554 (3)	0.31986 (9)	0.0315 (4)
C7	0.67576 (12)	1.0414 (3)	0.30768 (11)	0.0422 (4)
H7	0.6551	1.1672	0.2679	0.051*
C8	0.77135 (13)	1.0423 (4)	0.35423 (12)	0.0509 (5)
H8	0.8144	1.1693	0.3461	0.061*
C9	0.80266 (13)	0.8556 (4)	0.41245 (12)	0.0494 (5)
H9	0.8668	0.8566	0.4439	0.059*
C10	0.73927 (13)	0.6677 (3)	0.42410 (11)	0.0474 (5)
H10	0.7608	0.5399	0.4629	0.057*
C11	0.64353 (12)	0.6676 (3)	0.37835 (10)	0.0400 (4)
H11	0.6007	0.5404	0.3869	0.048*
C12	0.47214 (10)	1.0140 (3)	0.12240 (9)	0.0324 (4)
N1	0.43403 (9)	0.8032 (2)	0.32059 (8)	0.0319 (3)
N2	0.47887 (10)	1.0439 (2)	0.20808 (8)	0.0378 (3)
H2	0.4659	1.1869	0.2278	0.045*
N3	0.43973 (10)	1.2140 (3)	0.07345 (9)	0.0404 (4)
O1	0.46858 (9)	1.1527 (2)	0.40438 (7)	0.0448 (3)
O2	0.35735 (9)	0.4659 (2)	0.24816 (8)	0.0500 (3)
O3	0.49252 (9)	0.8126 (2)	0.09201 (7)	0.0461 (3)

H3A	0.4524 (13)	1.205 (3)	0.0176 (7)	0.060 (6)*
H3B	0.4387 (13)	1.371 (2)	0.0985 (9)	0.054 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0360 (9)	0.0376 (9)	0.0259 (8)	0.0048 (7)	0.0037 (7)	−0.0015 (7)
C2	0.0442 (10)	0.0562 (11)	0.0409 (10)	−0.0012 (8)	0.0166 (8)	−0.0043 (8)
C3	0.0367 (9)	0.0413 (9)	0.0505 (10)	0.0018 (7)	0.0134 (8)	0.0046 (8)
C4	0.0325 (9)	0.0344 (8)	0.0357 (9)	0.0040 (7)	−0.0005 (7)	−0.0001 (8)
C5	0.0394 (9)	0.0327 (8)	0.0265 (8)	0.0032 (7)	0.0097 (7)	−0.0020 (7)
C6	0.0352 (9)	0.0336 (8)	0.0277 (8)	0.0023 (7)	0.0114 (7)	−0.0028 (7)
C7	0.0463 (10)	0.0413 (10)	0.0413 (10)	−0.0002 (8)	0.0144 (8)	0.0030 (8)
C8	0.0407 (11)	0.0563 (11)	0.0585 (12)	−0.0110 (9)	0.0167 (9)	−0.0083 (10)
C9	0.0336 (9)	0.0679 (13)	0.0461 (11)	0.0056 (9)	0.0065 (8)	−0.0133 (10)
C10	0.0467 (11)	0.0560 (11)	0.0387 (10)	0.0143 (9)	0.0062 (8)	0.0045 (8)
C11	0.0399 (10)	0.0406 (9)	0.0406 (10)	0.0011 (7)	0.0108 (8)	0.0062 (8)
C12	0.0269 (8)	0.0441 (9)	0.0265 (9)	0.0002 (7)	0.0059 (6)	−0.0018 (7)
N1	0.0325 (7)	0.0373 (7)	0.0263 (7)	−0.0005 (6)	0.0068 (5)	−0.0048 (6)
N2	0.0518 (8)	0.0374 (8)	0.0243 (7)	0.0110 (6)	0.0079 (6)	−0.0018 (6)
N3	0.0483 (9)	0.0452 (9)	0.0280 (8)	0.0062 (7)	0.0086 (6)	0.0034 (7)
O1	0.0525 (7)	0.0405 (7)	0.0427 (7)	−0.0049 (5)	0.0128 (6)	−0.0095 (5)
O2	0.0503 (7)	0.0463 (7)	0.0516 (8)	−0.0043 (6)	0.0055 (6)	−0.0172 (6)
O3	0.0653 (8)	0.0462 (7)	0.0287 (6)	0.0074 (6)	0.0141 (6)	−0.0046 (5)

Geometric parameters (Å, °)

C1—O1	1.2064 (18)	C6—C11	1.384 (2)
C1—N1	1.3933 (19)	C7—C8	1.384 (3)
C1—C2	1.498 (2)	C7—H7	0.9300
C2—C3	1.514 (2)	C8—C9	1.374 (3)
C2—H2A	0.9700	C8—H8	0.9300
C2—H2B	0.9700	C9—C10	1.371 (3)
C3—C4	1.492 (2)	C9—H9	0.9300
C3—H3D	0.9700	C10—C11	1.381 (2)
C3—H3E	0.9700	C10—H10	0.9300
C4—O2	1.2139 (18)	C11—H11	0.9300
C4—N1	1.380 (2)	C12—O3	1.2364 (18)
C5—N2	1.4381 (19)	C12—N3	1.348 (2)
C5—N1	1.4703 (19)	C12—N2	1.3571 (19)
C5—C6	1.519 (2)	N2—H2	0.8600
C5—H5	0.9800	N3—H3A	0.940 (9)
C6—C7	1.379 (2)	N3—H3B	0.929 (9)
O1—C1—N1	123.52 (14)	C6—C7—C8	120.52 (16)
O1—C1—C2	128.73 (14)	C6—C7—H7	119.7
N1—C1—C2	107.72 (13)	C8—C7—H7	119.7
C1—C2—C3	105.34 (13)	C9—C8—C7	120.03 (17)
C1—C2—H2A	110.7	C9—C8—H8	120.0

C3—C2—H2A	110.7	C7—C8—H8	120.0
C1—C2—H2B	110.7	C10—C9—C8	119.93 (16)
C3—C2—H2B	110.7	C10—C9—H9	120.0
H2A—C2—H2B	108.8	C8—C9—H9	120.0
C4—C3—C2	105.34 (13)	C9—C10—C11	120.18 (17)
C4—C3—H3D	110.7	C9—C10—H10	119.9
C2—C3—H3D	110.7	C11—C10—H10	119.9
C4—C3—H3E	110.7	C10—C11—C6	120.44 (16)
C2—C3—H3E	110.7	C10—C11—H11	119.8
H3D—C3—H3E	108.8	C6—C11—H11	119.8
O2—C4—N1	123.70 (15)	O3—C12—N3	122.85 (14)
O2—C4—C3	128.05 (15)	O3—C12—N2	121.29 (14)
N1—C4—C3	108.25 (13)	N3—C12—N2	115.85 (14)
N2—C5—N1	110.08 (12)	C4—N1—C1	112.77 (13)
N2—C5—C6	115.35 (12)	C4—N1—C5	122.71 (12)
N1—C5—C6	111.24 (12)	C1—N1—C5	124.36 (12)
N2—C5—H5	106.5	C12—N2—C5	120.83 (13)
N1—C5—H5	106.5	C12—N2—H2	119.6
C6—C5—H5	106.5	C5—N2—H2	119.6
C7—C6—C11	118.88 (15)	C12—N3—H3A	113.7 (11)
C7—C6—C5	122.03 (14)	C12—N3—H3B	120.0 (10)
C11—C6—C5	118.91 (13)	H3A—N3—H3B	118.2 (12)
O1—C1—C2—C3	179.13 (16)	O2—C4—N1—C1	173.20 (15)
N1—C1—C2—C3	1.05 (17)	C3—C4—N1—C1	−7.39 (17)
C1—C2—C3—C4	−5.12 (17)	O2—C4—N1—C5	−2.4 (2)
C2—C3—C4—O2	−173.04 (16)	C3—C4—N1—C5	176.96 (13)
C2—C3—C4—N1	7.57 (17)	O1—C1—N1—C4	−174.25 (14)
N2—C5—C6—C7	−5.8 (2)	C2—C1—N1—C4	3.96 (17)
N1—C5—C6—C7	−132.05 (15)	O1—C1—N1—C5	1.3 (2)
N2—C5—C6—C11	179.07 (13)	C2—C1—N1—C5	179.52 (13)
N1—C5—C6—C11	52.79 (18)	N2—C5—N1—C4	103.21 (15)
C11—C6—C7—C8	−1.1 (2)	C6—C5—N1—C4	−127.66 (14)
C5—C6—C7—C8	−176.22 (15)	N2—C5—N1—C1	−71.93 (17)
C6—C7—C8—C9	0.7 (3)	C6—C5—N1—C1	57.21 (18)
C7—C8—C9—C10	0.3 (3)	O3—C12—N2—C5	−1.7 (2)
C8—C9—C10—C11	−1.0 (3)	N3—C12—N2—C5	176.62 (13)
C9—C10—C11—C6	0.6 (3)	N1—C5—N2—C12	−126.47 (14)
C7—C6—C11—C10	0.4 (2)	C6—C5—N2—C12	106.66 (15)
C5—C6—C11—C10	175.74 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.86	2.19	2.9600 (18)	149
N3—H3B···O3^{i}	0.929 (9)	2.489 (13)	3.2867 (19)	144.1 (13)
N3—H3A···O3^{ii}	0.940 (9)	2.037 (9)	2.9655 (17)	169.0 (16)

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*x*+1, −*y*+2, −*z*.

Fig. 1

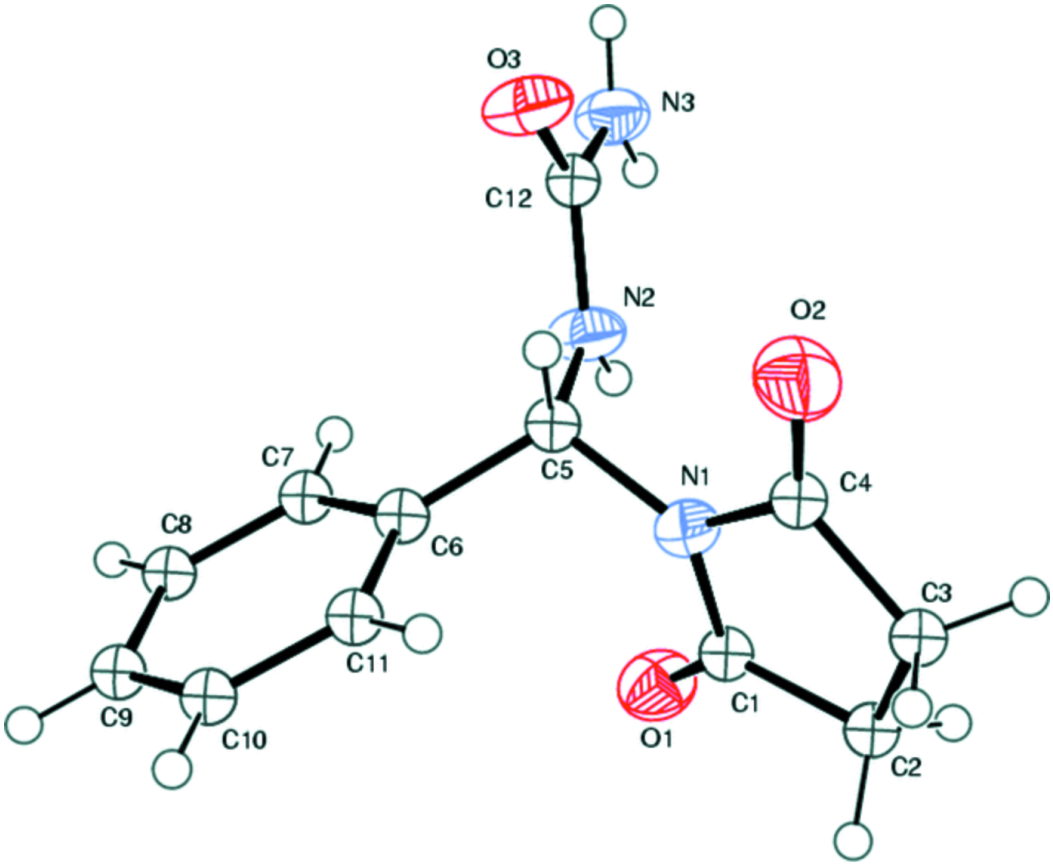

Fig. 2