organic compounds

1*H*-Pyrrole-2-carbohydrazide

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The title compound, $C_5H_7N_3O$, was obtained by the reaction of ethyl 1*H*-pyrrol-2-carboxylate and hydrazide hydrate. In the crystal, molecules are linked *via* intermolecular N—H···N and N—H···O hydrogen bonds, forming a supramolecular grid.

Related literature

For background to pyrrole derivatives and their biological activity, see: Joshi *et al.* (2008); Demirayak *et al.* (1999); Halazy & Magnus (1984); Bijev (2006); Sbardella *et al.* (2004).
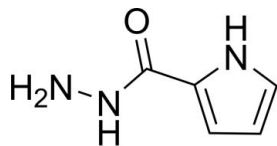
Experimental

Crystal data

$C_5H_7N_3O$
M_r = 125.14
Orthorhombic, *Pbca*
a = 9.9789 (16) Å
b = 8.5633 (14) Å
c = 13.657 (2) Å

V = 1167.0 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
$0.31 \times 0.28 \times 0.16$ mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.968, T_{max} = 0.983

5327 measured reflections
1043 independent reflections
758 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.146
S = 1.04
1043 reflections
90 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N3^i	0.86	2.15	2.996 (2)	169
N2—H2···O1^{ii}	0.86	2.06	2.8422 (19)	151
N3—H3*B*···O1^{iii}	0.91 (3)	2.12 (3)	3.023 (3)	168 (2)

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 1$; (iii) $-x + 1, -y + 1, -z + 1$.

Data collection: *APEX2* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; 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: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FY2001).

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supplementary materials

1*H*-Pyrrole-2-carbohydrazide

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Comment

Pyrrole is one of the most ubiquitous heterocycles in the plant and animal kingdom because of its participation as a subunit of chlorophyll in plant cells and hemin and vitamin B12 in animal cells (Joshi *et al.*, 2008). Pyrrole and its derivatives have shown to possess biological activities such as antibacterial (Demirayak *et al.*, 1999), antitumor (Halazy *et al.*, 1984), analgesics, antitubercular (Bijev, 2006), anti-inflammatory, and antiallergic (Sbardella *et al.*, 2004). Several macromolecular antibiotics having pyrrole structure were isolated from biological sources and their activities were defined.

The molecular structure for 2-pyrrole hydrazide is shown in Fig. 1. The crystal structure is stabilized by N1—H1⋯N3, N2—H2⋯O1 and N3—H3B⋯O1 hydrogen bonds, as shown in Fig. 2 and Table 1.

In addition, as shown in Fig.3, the packing diagram of the title compound looks like the wave viewed down the *a* axis.

Experimental

To a 25 mL round-bottomed flask equipped with a magnetic stirrer, 0.5 mL of hydrazine hydrate (80% in water) and 0.1392 g of 1*H*-pyrrol-2-carboxlic acid ethyl ester (1 mmol) were added. Then the temperature of the mixture was elevated to 70°C for 45 min and the mixture was cooled to room temperature. The formed suspension was filtered off, washed with Et$_2$O, and recrystallized from absolute ethyl alcohol. 0.113 g of the hydrazide was obtained with a yield of 90%.

Refinement

H atoms attached to N3 were located in a difference Fourier map. All other H atoms were placed at calculated positions and all were refined in riding model, with N—H and C—H distances in the range of 0.86 and 0.93 Å and U_{iso}(H)= 1.2 U_{eq} of the attached N and C atoms.
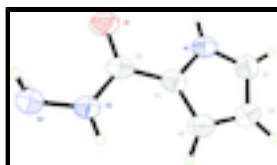
Figures

Fig. 1. The molecular structure for 2-pyrrole hydrazide, with atom labels and 50% probability displacement ellipsoids for non-H atoms.
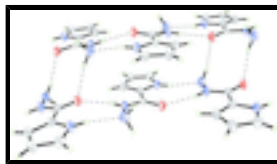

Fig. 2. View of the hydrogen bonds for 2-pyrrole hydrazide, H atoms not involved in hydrogen bonding have been omitted.
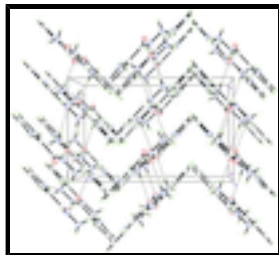
supplementary materials

Fig. 3. The packing for 2-pyrrole hydrazide, viewed down the *a* axis.

1*H*-Pyrrole-2-carbohydrazide

Crystal data

$C_5H_7N_3O$	$D_x = 1.424$ Mg m^{-3}
$M_r = 125.14$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Orthorhombic, *Pbca*	Cell parameters from 990 reflections
$a = 9.9789$ (16) Å	$\theta = 3.0–22.4°$
$b = 8.5633$ (14) Å	$\mu = 0.11$ mm^{-1}
$c = 13.657$ (2) Å	$T = 296$ K
$V = 1167.0$ (3) Å^3	Block, colourless
$Z = 8$	$0.31 \times 0.28 \times 0.16$ mm
$F(000) = 528$	

Data collection

Bruker APEXII CCD diffractometer	1043 independent reflections
Radiation source: fine-focus sealed tube	758 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.031$
φ and ω scans	$\theta_{max} = 25.1°$, $\theta_{min} = 3.0°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)	$h = -11 \rightarrow 11$
$T_{min} = 0.968$, $T_{max} = 0.983$	$k = -6 \rightarrow 10$
5327 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.043$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.146$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0957P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
1043 reflections	$(\Delta/\sigma)_{max} < 0.001$
90 parameters	$\Delta\rho_{max} = 0.16$ e Å^{-3}

0 restraints $\Delta\rho_{min} = -0.21$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	$U_{iso}*/U_{eq}$
N1	0.55417 (12)	0.04787 (18)	0.64606 (11)	0.0469 (5)
H1	0.6352	0.0752	0.6332	0.056*
N2	0.34109 (14)	0.28224 (17)	0.48978 (11)	0.0495 (5)
H2	0.2667	0.2414	0.5087	0.059*
N3	0.33807 (16)	0.3955 (2)	0.41573 (15)	0.0551 (5)
O1	0.56366 (11)	0.29286 (15)	0.50912 (10)	0.0538 (5)
C1	0.5178 (2)	−0.0654 (2)	0.71020 (14)	0.0523 (5)
H1A	0.5761	−0.1266	0.7470	0.063*
C2	0.3816 (2)	−0.0744 (2)	0.71164 (14)	0.0544 (6)
H2A	0.3304	−0.1421	0.7495	0.065*
C3	0.33310 (17)	0.0371 (2)	0.64571 (14)	0.0510 (6)
H3	0.2435	0.0573	0.6318	0.061*
C4	0.44238 (15)	0.1116 (2)	0.60516 (13)	0.0418 (5)
C5	0.45432 (16)	0.2352 (2)	0.53214 (13)	0.0423 (5)
H3A	0.394 (3)	0.359 (3)	0.3665 (19)	0.086 (8)*
H3B	0.380 (3)	0.483 (3)	0.4387 (19)	0.095 (9)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0320 (8)	0.0562 (10)	0.0526 (10)	−0.0009 (7)	0.0010 (6)	0.0026 (8)
N2	0.0285 (8)	0.0569 (10)	0.0631 (10)	−0.0010 (6)	0.0003 (6)	0.0119 (8)
N3	0.0383 (10)	0.0595 (12)	0.0674 (12)	−0.0003 (8)	−0.0021 (8)	0.0121 (10)
O1	0.0304 (8)	0.0574 (9)	0.0737 (10)	−0.0019 (5)	0.0016 (6)	0.0054 (7)
C1	0.0483 (11)	0.0604 (12)	0.0481 (11)	0.0021 (9)	−0.0002 (8)	0.0056 (10)
C2	0.0455 (12)	0.0631 (13)	0.0545 (12)	−0.0060 (9)	0.0063 (9)	0.0041 (10)
C3	0.0357 (10)	0.0618 (12)	0.0554 (12)	−0.0037 (9)	0.0001 (8)	−0.0008 (10)
C4	0.0336 (9)	0.0469 (11)	0.0447 (10)	−0.0001 (7)	−0.0003 (7)	−0.0048 (8)
C5	0.0319 (10)	0.0452 (10)	0.0497 (11)	0.0003 (8)	0.0005 (7)	−0.0091 (9)

Geometric parameters (Å, °)

N1—C1	1.356 (2)	O1—C5	1.2380 (18)
N1—C4	1.362 (2)	C1—C2	1.361 (3)
N1—H1	0.8600	C1—H1A	0.9300
N2—C5	1.332 (2)	C2—C3	1.399 (3)
N2—N3	1.402 (2)	C2—H2A	0.9300
N2—H2	0.8600	C3—C4	1.380 (2)
N3—H3A	0.93 (3)	C3—H3	0.9300
N3—H3B	0.91 (3)	C4—C5	1.459 (3)
C1—N1—C4	109.41 (15)	C1—C2—C3	107.33 (17)
C1—N1—H1	125.3	C1—C2—H2A	126.3
C4—N1—H1	125.3	C3—C2—H2A	126.3
C5—N2—N3	122.74 (15)	C4—C3—C2	107.48 (16)
C5—N2—H2	118.6	C4—C3—H3	126.3
N3—N2—H2	118.6	C2—C3—H3	126.3
N2—N3—H3A	106.1 (15)	N1—C4—C3	107.30 (17)
N2—N3—H3B	108.1 (17)	N1—C4—C5	120.28 (14)
H3A—N3—H3B	104 (2)	C3—C4—C5	132.42 (15)
N1—C1—C2	108.47 (17)	O1—C5—N2	121.13 (18)
N1—C1—H1A	125.8	O1—C5—C4	122.32 (15)
C2—C1—H1A	125.8	N2—C5—C4	116.54 (15)
C4—N1—C1—C2	−0.5 (2)	N3—N2—C5—O1	1.6 (3)
N1—C1—C2—C3	0.2 (2)	N3—N2—C5—C4	−177.42 (17)
C1—C2—C3—C4	0.2 (2)	N1—C4—C5—O1	−4.4 (3)
C1—N1—C4—C3	0.6 (2)	C3—C4—C5—O1	175.93 (18)
C1—N1—C4—C5	−179.09 (15)	N1—C4—C5—N2	174.58 (16)
C2—C3—C4—N1	−0.5 (2)	C3—C4—C5—N2	−5.1 (3)
C2—C3—C4—C5	179.19 (19)		

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N3[i]	0.86	2.15	2.996 (2)	169
N2—H2···O1[ii]	0.86	2.06	2.8422 (19)	151
N3—H3B···O1[iii]	0.91 (3)	2.12 (3)	3.023 (3)	168 (2)

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

Fig. 1

Fig. 2

Fig. 3