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5,5′-(1-Phenylethane-1,1-diyl)bis(1*H*-pyrrole-2-carboxaldehyde)

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Keywords: crystal structure; pyrrole; hydrogen bonding; Hirshfeld surface analysis.

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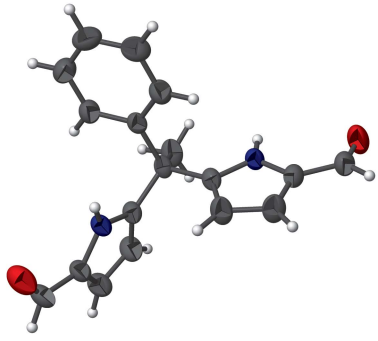
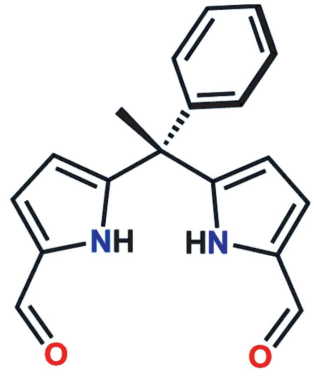
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{18}H_{16}N_2O_2$, the dihedral angle between the pyrrole rings is 79.47 (9)°, with the N—H groups approximately orthogonal (H—N···N—H pseudo torsion angle = −106°). In the crystal, N—H···O hydrogen bonds link the molecules into [11$\bar{1}$] chains. A C—H···O interaction is also observed.

3D view



Chemical scheme



Structure description

Dipyrromethane-dialdehydes are intermediates for the preparation of various macrocyclic (Love *et al.*, 2003) and acyclic diiminodipyrromethane Schiff bases (*e.g.*: Deliomeroglu *et al.*, 2016). As a part of our studies in this area, we now report the crystal structure of the title compound.

The dihedral angles between the N1-pyrrole ring (*A*), N2-pyrrole ring (*B*) and the C13–C18 phenyl ring (*C*) are *A*/*B* = 79.47 (9), *A*/*C* = 87.21 (8) and *B*/*C* = 70.40 (8)°. The N1—C6—C7—O1 [−2.7 (2)°] and N2—C11—C12—O2 [2.8 (2)°] torsion angles indicate that the aldehyde groups are almost coplanar with their adjacent pyrrole ring systems (Fig. 1).

In the crystal, N—H···O hydrogen bonds (Table 1) link the molecules into [11$\bar{1}$] chains (Fig. 2). A weak C—H···O hydrogen bond consolidates the chains. The Hirshfeld surface and two-dimensional fingerprint plots were generated with *CrystalExplorer17.5* (Turner *et al.*, 2017) (see supplementary materials). The percentage contributions from the different interatomic contacts to the Hirshfeld surfaces are as follows: H···H (47.9%), C···H/H···C (27.5%), O···H/H···O (21.5%), N···H/H···N (1.5%) and C···O/O···C (1.5%).

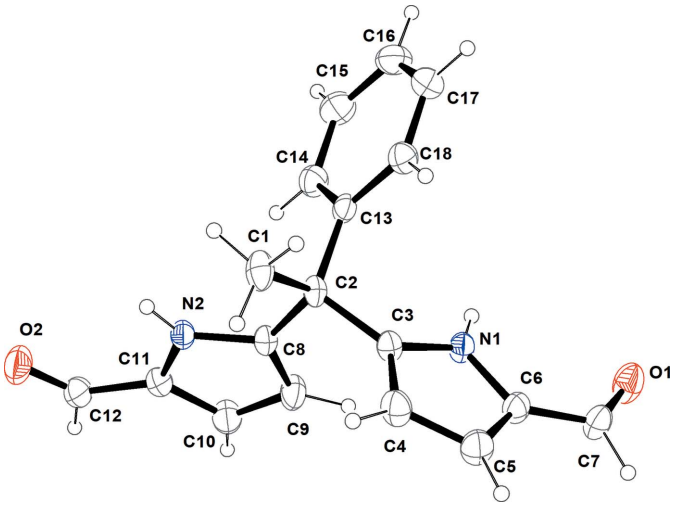


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

Synthesis and crystallization

The title compound, $C_{18}H_{16}N_2O_2$ was prepared by the reported method (Fig. 3); Muwal *et al.* 2018) and colourless cubes were recrystallized from a toluene–hexane solvent mixture at −4°C.

Refinement

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

Acknowledgements

This work was supported by the VFSTR Deemed to be University, Vadlamudi, Andhra Pradesh, India, under the scheme of seed grant for research faculties. SKL and EP thanks to VFSTR for providing facilities and fellowships.

Funding information

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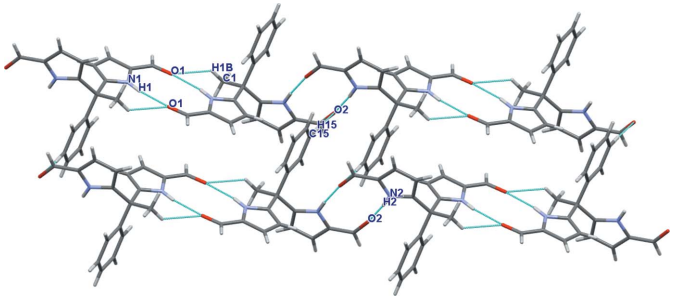

Figure 2
Illustration of the different hydrogen bonds (N—H···O and C—H···O) in the title compound viewed along [001]. Hydrogen bonds are drawn as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.08	2.9321 (16)	171
N2—H2···O2^{ii}	0.86	2.03	2.8838 (15)	173
C1—H1B···O1^{i}	0.96	2.58	3.3419 (18)	137

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{16}N_2O_2$
M_r	292.33
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, b, c (Å)	7.0679 (2), 9.6856 (4), 12.1510 (5)
α, β, γ (°)	101.560 (3), 93.302 (3), 111.270 (4)
V (Å^3)	751.66 (5)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.18 × 0.15 × 0.13
Data collection	
Diffractometer	XtaLAB Pro: Kappa dual offset/far
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.901, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13274, 4208, 3307
R_{int}	0.022
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.726
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.142, 1.03
No. of reflections	4208
No. of parameters	200
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.32, −0.19

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT2014/5* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), , *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008).

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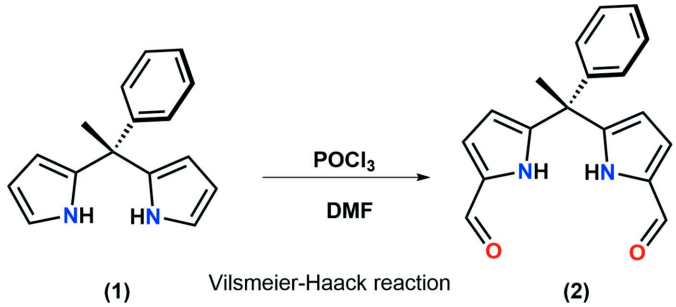

Figure 3
Reaction scheme.

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
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full crystallographic data

IUCrData (2019). **4**, x191660 [https://doi.org/10.1107/S2414314619016602]

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Crystal data

$C_{18}H_{16}N_2O_2$
M_r = 292.33
Triclinic, $P\overline{1}$
a = 7.0679 (2) Å
b = 9.6856 (4) Å
c = 12.1510 (5) Å
α = 101.560 (3)°
β = 93.302 (3)°
γ = 111.270 (4)°
V = 751.66 (5) Å^3
Z = 2
$F(000)$ = 308
D_x = 1.292 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5094 reflections
θ = 3.7–29.9°
μ = 0.09 mm^{-1}
T = 293 K
Cube, colourless
0.18 × 0.15 × 0.13 mm

Data collection

XtaLAB Pro: Kappa dual offset/far diffractometer
Radiation source: fine-focus sealed X-ray tube
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2015)
T_{min} = 0.901, T_{max} = 1.000
13274 measured reflections
4208 independent reflections
3307 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 31.1°, θ_{min} = 3.1°
h = −9→10
k = −13→13
l = −17→17
Standard reflections: see text; every none reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.142
S = 1.03
4208 reflections
200 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0767P)^2 + 0.1364P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.69498 (16)	0.13760 (11)	0.87005 (8)	0.0348 (2)
H1	0.592279	0.131280	0.906260	0.042*
N2	0.94852 (16)	0.52913 (11)	0.69220 (8)	0.0350 (2)
H2	0.897693	0.470214	0.625766	0.042*
O2	1.18939 (19)	0.67425 (13)	0.53044 (9)	0.0593 (3)
O1	0.63340 (19)	−0.10500 (15)	0.98826 (12)	0.0649 (3)
C8	0.87280 (19)	0.50458 (13)	0.78942 (10)	0.0349 (3)
C13	0.53349 (18)	0.30398 (14)	0.68621 (10)	0.0346 (3)
C6	0.8011 (2)	0.04229 (15)	0.86453 (11)	0.0399 (3)
C3	0.77809 (19)	0.24329 (14)	0.80925 (10)	0.0347 (3)
C2	0.69195 (19)	0.36169 (13)	0.79432 (10)	0.0338 (2)
C11	1.1197 (2)	0.66317 (14)	0.71604 (11)	0.0398 (3)
C14	0.4611 (2)	0.40434 (16)	0.64815 (12)	0.0445 (3)
H14	0.512212	0.506479	0.686836	0.053*
C18	0.4538 (2)	0.15255 (16)	0.62642 (12)	0.0452 (3)
H18	0.500715	0.083660	0.650140	0.054*
C7	0.7592 (2)	−0.07648 (17)	0.92390 (13)	0.0471 (3)
H7	0.835458	−0.137403	0.912050	0.057*
C12	1.2317 (2)	0.72554 (15)	0.63178 (13)	0.0464 (3)
H12	1.348509	0.814225	0.657358	0.056*
C9	0.9961 (2)	0.62497 (16)	0.87798 (11)	0.0470 (3)
H9	0.978635	0.637712	0.954118	0.056*
C5	0.9541 (2)	0.09002 (19)	0.79891 (13)	0.0505 (3)
H5	1.049116	0.046264	0.780729	0.061*
C1	0.5846 (3)	0.40046 (18)	0.89673 (12)	0.0478 (3)
H1A	0.677390	0.429200	0.965753	0.072*
H1B	0.465129	0.312760	0.898067	0.072*
H1C	0.544689	0.483480	0.889915	0.072*
C10	1.1508 (2)	0.72322 (16)	0.83195 (12)	0.0500 (3)
H10	1.255985	0.812989	0.871959	0.060*
C4	0.9407 (2)	0.21604 (18)	0.76469 (13)	0.0495 (3)
H4	1.025661	0.271584	0.720018	0.059*
C15	0.3139 (2)	0.3539 (2)	0.55333 (14)	0.0543 (4)
H15	0.267142	0.422408	0.528760	0.065*
C17	0.3053 (2)	0.10196 (19)	0.53181 (15)	0.0574 (4)
H17	0.252517	−0.000256	0.493242	0.069*
C16	0.2361 (2)	0.2028 (2)	0.49507 (15)	0.0587 (4)
H16	0.137392	0.169268	0.431307	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0370 (5)	0.0360 (5)	0.0340 (5)	0.0132 (4)	0.0090 (4)	0.0142 (4)
N2	0.0403 (5)	0.0302 (5)	0.0283 (5)	0.0065 (4)	0.0053 (4)	0.0066 (4)
O2	0.0674 (7)	0.0533 (6)	0.0445 (6)	0.0066 (5)	0.0180 (5)	0.0121 (5)

O1	0.0602 (7)	0.0681 (7)	0.0862 (9)	0.0277 (6)	0.0297 (6)	0.0500 (7)
C8	0.0428 (6)	0.0322 (5)	0.0298 (5)	0.0134 (5)	0.0067 (4)	0.0090 (4)
C13	0.0334 (5)	0.0372 (6)	0.0361 (6)	0.0122 (5)	0.0132 (4)	0.0154 (5)
C6	0.0428 (6)	0.0395 (6)	0.0410 (6)	0.0172 (5)	0.0057 (5)	0.0147 (5)
C3	0.0404 (6)	0.0355 (6)	0.0300 (5)	0.0135 (5)	0.0095 (4)	0.0126 (4)
C2	0.0419 (6)	0.0336 (5)	0.0304 (5)	0.0157 (5)	0.0130 (4)	0.0129 (4)
C11	0.0420 (6)	0.0316 (6)	0.0398 (6)	0.0069 (5)	0.0042 (5)	0.0090 (5)
C14	0.0451 (7)	0.0405 (7)	0.0518 (8)	0.0176 (6)	0.0084 (6)	0.0168 (6)
C18	0.0481 (7)	0.0379 (6)	0.0485 (7)	0.0141 (6)	0.0066 (6)	0.0124 (5)
C7	0.0457 (7)	0.0445 (7)	0.0589 (8)	0.0201 (6)	0.0090 (6)	0.0237 (6)
C12	0.0457 (7)	0.0344 (6)	0.0493 (8)	0.0031 (5)	0.0105 (6)	0.0106 (5)
C9	0.0616 (8)	0.0420 (7)	0.0301 (6)	0.0133 (6)	0.0037 (6)	0.0057 (5)
C5	0.0530 (8)	0.0583 (9)	0.0560 (8)	0.0320 (7)	0.0202 (6)	0.0246 (7)
C1	0.0636 (9)	0.0518 (8)	0.0415 (7)	0.0306 (7)	0.0270 (6)	0.0191 (6)
C10	0.0560 (8)	0.0386 (7)	0.0404 (7)	0.0050 (6)	−0.0015 (6)	0.0036 (5)
C4	0.0522 (8)	0.0569 (8)	0.0554 (8)	0.0276 (7)	0.0270 (6)	0.0303 (7)
C15	0.0474 (8)	0.0620 (9)	0.0625 (9)	0.0251 (7)	0.0064 (7)	0.0270 (8)
C17	0.0521 (8)	0.0481 (8)	0.0589 (9)	0.0100 (7)	0.0009 (7)	0.0036 (7)
C16	0.0433 (8)	0.0718 (11)	0.0548 (9)	0.0165 (7)	−0.0016 (6)	0.0143 (8)

Geometric parameters (Å, º)

N1—C3	1.3622 (14)	C14—C15	1.384 (2)
N1—C6	1.3793 (16)	C14—H14	0.9300
N1—H1	0.8600	C18—C17	1.388 (2)
N2—C8	1.3533 (15)	C18—H18	0.9300
N2—C11	1.3795 (16)	C7—H7	0.9300
N2—H2	0.8600	C12—H12	0.9300
O2—C12	1.2071 (18)	C9—C10	1.394 (2)
O1—C7	1.2113 (18)	C9—H9	0.9300
C8—C9	1.3888 (18)	C5—C4	1.398 (2)
C8—C2	1.5202 (16)	C5—H5	0.9300
C13—C18	1.3867 (19)	C1—H1A	0.9600
C13—C14	1.3907 (18)	C1—H1B	0.9600
C13—C2	1.5410 (17)	C1—H1C	0.9600
C6—C5	1.3798 (19)	C10—H10	0.9300
C6—C7	1.4288 (18)	C4—H4	0.9300
C3—C4	1.3850 (18)	C15—C16	1.380 (3)
C3—C2	1.5152 (17)	C15—H15	0.9300
C2—C1	1.5470 (16)	C17—C16	1.374 (3)
C11—C10	1.3833 (19)	C17—H17	0.9300
C11—C12	1.4261 (18)	C16—H16	0.9300
C3—N1—C6	109.44 (10)	O1—C7—C6	125.90 (14)
C3—N1—H1	125.3	O1—C7—H7	117.0
C6—N1—H1	125.3	C6—C7—H7	117.0
C8—N2—C11	109.55 (10)	O2—C12—C11	126.24 (13)
C8—N2—H2	125.2	O2—C12—H12	116.9

C11—N2—H2	125.2	C11—C12—H12	116.9
N2—C8—C9	107.91 (11)	C8—C9—C10	107.53 (12)
N2—C8—C2	123.11 (10)	C8—C9—H9	126.2
C9—C8—C2	128.86 (11)	C10—C9—H9	126.2
C18—C13—C14	118.02 (12)	C6—C5—C4	107.65 (12)
C18—C13—C2	122.18 (11)	C6—C5—H5	126.2
C14—C13—C2	119.76 (11)	C4—C5—H5	126.2
N1—C6—C5	107.49 (11)	C2—C1—H1A	109.5
N1—C6—C7	123.62 (12)	C2—C1—H1B	109.5
C5—C6—C7	128.80 (13)	H1A—C1—H1B	109.5
N1—C3—C4	107.70 (11)	C2—C1—H1C	109.5
N1—C3—C2	122.84 (10)	H1A—C1—H1C	109.5
C4—C3—C2	129.42 (11)	H1B—C1—H1C	109.5
C3—C2—C8	106.88 (10)	C11—C10—C9	107.68 (12)
C3—C2—C13	111.23 (10)	C11—C10—H10	126.2
C8—C2—C13	111.41 (9)	C9—C10—H10	126.2
C3—C2—C1	110.70 (9)	C3—C4—C5	107.72 (12)
C8—C2—C1	108.65 (10)	C3—C4—H4	126.1
C13—C2—C1	107.96 (11)	C5—C4—H4	126.1
N2—C11—C10	107.32 (11)	C16—C15—C14	120.39 (15)
N2—C11—C12	123.89 (12)	C16—C15—H15	119.8
C10—C11—C12	128.73 (13)	C14—C15—H15	119.8
C15—C14—C13	120.75 (14)	C16—C17—C18	120.05 (15)
C15—C14—H14	119.6	C16—C17—H17	120.0
C13—C14—H14	119.6	C18—C17—H17	120.0
C13—C18—C17	121.17 (14)	C17—C16—C15	119.60 (15)
C13—C18—H18	119.4	C17—C16—H16	120.2
C17—C18—H18	119.4	C15—C16—H16	120.2

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.08	2.9321 (16)	171
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