organic compounds

N,N′-Bis(pyridin-3-yl)terephthalamide–terephthalic acid (1/1)

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In the title compound, $C_{18}H_{14}N_4O_2 \cdot C_8H_6O_4$, both types of molecule lie on inversion centers. In the N,N′-bis(pyridin-3-yl)terephthalamide molecule, the pyridine ring forms a dihedral angle of 11.33 (9)° with the central benzene ring. In the crystal, N—H⋯O and O—H⋯N hydrogen bonds connect the components into a three-dimensional network.

Related literature

For related structures, see: Xiao *et al.* (2011), Wang *et al.* (2009).
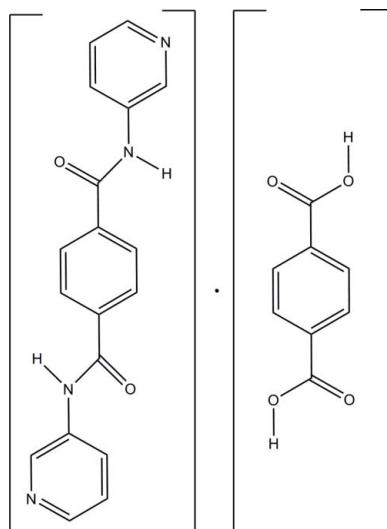
Experimental

Crystal data

$C_{18}H_{14}N_4O_2 \cdot C_8H_6O_4$
$M_r = 484.46$
Monoclinic, $P2_1/c$
$a = 11.0001$ (3) Å
$b = 10.8080$ (2) Å
$c = 9.6903$ (2) Å
$\beta = 106.830$ (2)°

$V = 1102.73$ (4) Å^3
$Z = 2$
Mo Kα radiation
$\mu = 0.11$ mm^{-1}
$T = 296$ K
$0.25 \times 0.24 \times 0.22$ mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (SABADS; Sheldrick, 1996)
$T_{min} = 0.974$, $T_{max} = 0.977$

8117 measured reflections
1939 independent reflections
1640 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.095$
$S = 1.07$
1939 reflections
171 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2N⋯O1[i]	0.89 (2)	1.98 (2)	2.8616 (18)	171.3 (18)
O2—H1N⋯N1[ii]	1.00 (3)	1.69 (3)	2.6938 (19)	178 (2)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5338).

References

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Wang, S., Yang, T., Li, Z. & Yu, X. (2009). *Acta Cryst.* E**65**, o2198.
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supporting information

N,*N*′-Bis(pyridin-3-yl)terephthalamide–terephthalic acid (1/1)

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S1. Comment

Pyridine amide derivatives and carboxylic acids easily form hydrogen bonds therefore they are useful to construct supramoleculur structures (e.g. Xiao *et al.*, 2011; Wang *et al.*, 2009). Herein, we use N,N′-di(pyridin-3-yl)terephthalamide and terephthalic acid to construct a supramolecular compound. The crystal structure of the title compound is presented herein.

  The molecular structure of the title compound is shown in Fig. 1. The symmetry unique pyridine ring forms a dihedral angle of 11.33 (9)° with the central benzene ring. In the crystal, N—H···O and O—H···N hydrogen bonds connect the components of the structure into a three dimensional network (Fig. 2).

S2. Experimental

N,N′-di(pyridin-3-yl)terephthalamide (0.2 mmol) and terephthalic acid (0.2 mmol) was sealed in a teflon reactor with 6 mL water, and heated at 433 K for 2 days, and then cooled to room temperature. The single crystals were obtained by slow evaporation.

S3. Refinement

H atoms bonded to C atoms were placed in calculated positions with C—H = 0.93Å and included using a riding-model approximation with $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms bonded to O and N atoms were refined independently with isotropic displacement parameters.
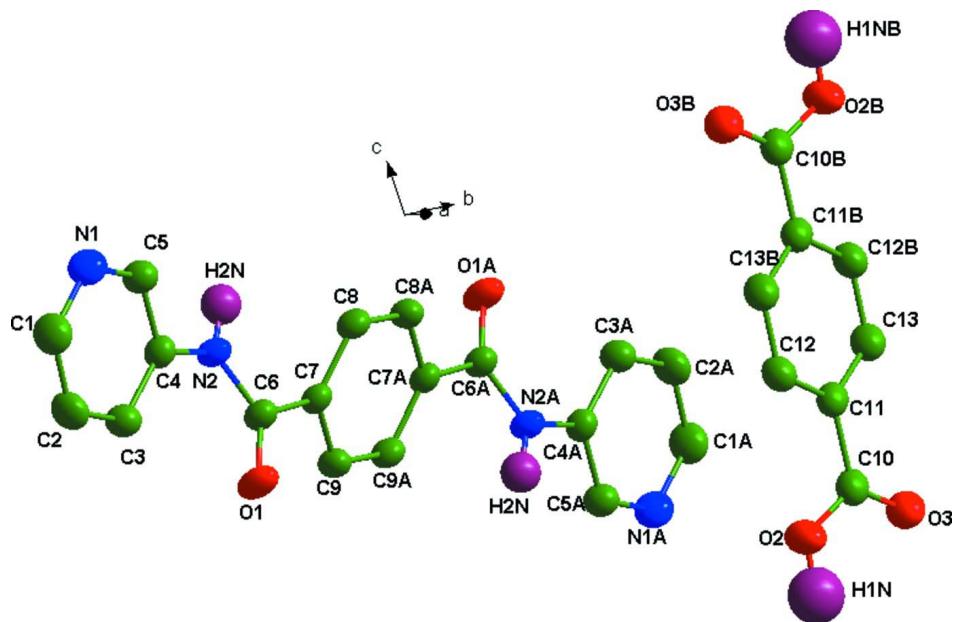
Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level (symmetry code; (A) -x + 1, -y + 1, -z + 1; (B) -x + 2, -y + 2, -z + 1). Hydrogen atoms bonded to C atoms are not shown.
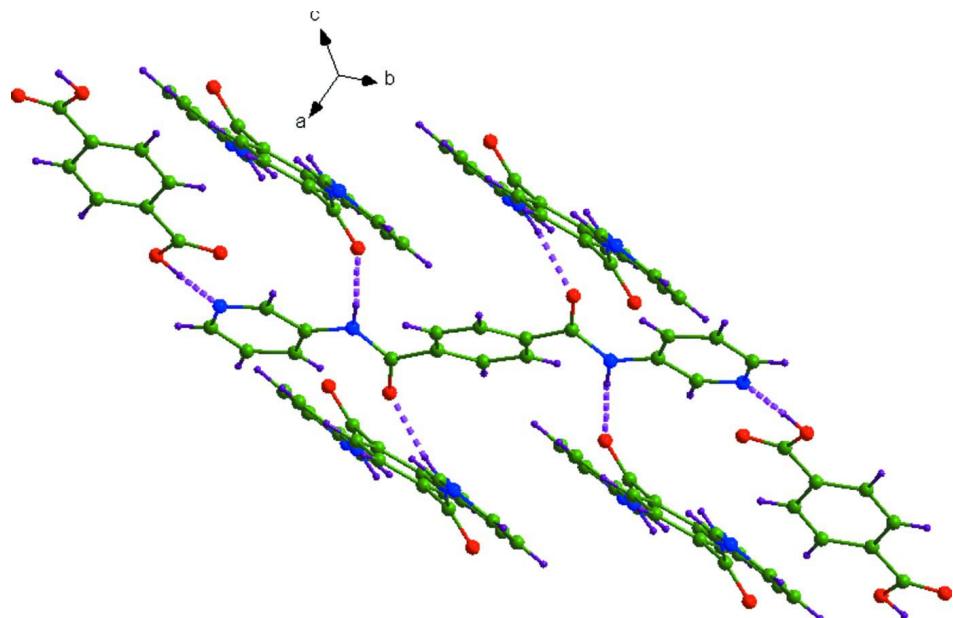
Figure 2

Part of the crystal structure with hydrogen bonds shown as pink dashed lines. H atoms are purple.

N,N′-Bis(pyridin-3-yl)terephthalamide; terephthalic acid

Crystal data
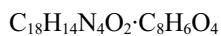
$C_{18}H_{14}N_4O_2·C_8H_6O_4$
M_r = 484.46
Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc
a = 11.0001 (3) Å
b = 10.8080 (2) Å

$c = 9.6903$ (2) Å

$\beta = 106.830$ (2)°

$V = 1102.73$ (4) Å^3

$Z = 2$

$F(000) = 504$

$D_x = 1.459$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2279 reflections

$\theta = 2.7–25.2°$

$\mu = 0.11$ mm^{-1}

$T = 296$ K

Block, colourless

$0.25 \times 0.24 \times 0.22$ mm

Data collection

Bruker SMART CCD
   diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
   (SABADS; Sheldrick, 1996)

$T_{min} = 0.974$, $T_{max} = 0.977$

8117 measured reflections

1939 independent reflections

1640 reflections with $I > 2\sigma(I)$

$R_{int} = 0.035$

$\theta_{max} = 25.0°$, $\theta_{min} = 1.9°$

$h = -13\rightarrow12$

$k = -12\rightarrow11$

$l = -11\rightarrow11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.095$

$S = 1.07$

1939 reflections

171 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 atoms treated by a mixture of independent
   and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0378P)^2 + 0.3019P]$
   where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.14$ e Å^{-3}

$\Delta\rho_{min} = -0.19$ 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}
C10	0.91350 (16)	1.01651 (17)	0.18960 (19)	0.0385 (4)
C11	0.95943 (16)	1.00643 (16)	0.35009 (18)	0.0358 (4)
C8	0.58897 (16)	0.45790 (15)	0.62372 (17)	0.0336 (4)
H8	0.6489	0.4302	0.7068	0.040*
C7	0.54563 (16)	0.37897 (14)	0.50665 (16)	0.0308 (4)
C5	0.74809 (16)	0.04149 (16)	0.79727 (18)	0.0367 (4)
H5	0.7580	0.1028	0.8671	0.044*
C3	0.66694 (17)	−0.02113 (16)	0.55212 (18)	0.0381 (4)
H3	0.6219	−0.0058	0.4565	0.046*

C6	0.59441 (16)	0.25022 (15)	0.50444 (17)	0.0339 (4)
C9	0.45681 (17)	0.42247 (15)	0.38296 (17)	0.0351 (4)
H9	0.4279	0.3704	0.3038	0.042*
C12	1.01793 (16)	0.89971 (16)	0.41856 (19)	0.0388 (4)
H12	1.0304	0.8326	0.3642	0.047*
C4	0.68076 (15)	0.06989 (15)	0.65646 (17)	0.0313 (4)
C13	0.94247 (17)	1.10642 (16)	0.43264 (19)	0.0399 (4)
H13	0.9041	1.1782	0.3874	0.048*
C2	0.72163 (18)	−0.13483 (16)	0.5939 (2)	0.0429 (5)
H2	0.7154	−0.1971	0.5259	0.051*
C1	0.78552 (18)	−0.15624 (17)	0.7362 (2)	0.0460 (5)
H1	0.8204	−0.2341	0.7630	0.055*
N1	0.79926 (14)	−0.06916 (14)	0.83764 (15)	0.0423 (4)
N2	0.62725 (14)	0.18931 (13)	0.63090 (14)	0.0338 (3)
H2N	0.6221 (18)	0.2299 (18)	0.709 (2)	0.048 (5)*
O1	0.60228 (13)	0.20383 (11)	0.39151 (12)	0.0476 (4)
O2	0.91449 (14)	0.91030 (12)	0.12264 (15)	0.0524 (4)
H1N	0.874 (3)	0.918 (3)	0.016 (3)	0.095 (9)*
O3	0.87627 (13)	1.11288 (12)	0.12828 (13)	0.0501 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C10	0.0381 (9)	0.0349 (10)	0.0398 (10)	−0.0038 (8)	0.0069 (8)	0.0027 (8)
C11	0.0364 (9)	0.0332 (10)	0.0353 (9)	−0.0037 (7)	0.0063 (7)	0.0034 (7)
C8	0.0435 (9)	0.0310 (9)	0.0255 (8)	0.0035 (7)	0.0086 (7)	0.0045 (7)
C7	0.0437 (9)	0.0256 (9)	0.0255 (8)	0.0017 (7)	0.0136 (7)	0.0033 (7)
C5	0.0458 (10)	0.0313 (10)	0.0321 (9)	0.0041 (8)	0.0100 (8)	0.0008 (7)
C3	0.0485 (10)	0.0328 (10)	0.0317 (9)	0.0024 (8)	0.0094 (8)	−0.0004 (7)
C6	0.0472 (10)	0.0294 (9)	0.0273 (9)	0.0014 (7)	0.0143 (7)	0.0014 (7)
C9	0.0514 (10)	0.0279 (9)	0.0258 (8)	0.0002 (7)	0.0109 (8)	−0.0011 (7)
C12	0.0434 (10)	0.0313 (9)	0.0388 (10)	−0.0002 (8)	0.0072 (8)	−0.0022 (8)
C4	0.0384 (9)	0.0259 (9)	0.0311 (9)	0.0028 (7)	0.0123 (7)	0.0026 (7)
C13	0.0430 (10)	0.0312 (10)	0.0408 (10)	0.0023 (8)	0.0047 (8)	0.0036 (8)
C2	0.0539 (11)	0.0282 (10)	0.0452 (11)	0.0045 (8)	0.0122 (9)	−0.0059 (8)
C1	0.0530 (11)	0.0311 (10)	0.0509 (12)	0.0092 (8)	0.0105 (9)	0.0034 (9)
N1	0.0493 (9)	0.0362 (9)	0.0377 (8)	0.0086 (7)	0.0066 (7)	0.0049 (7)
N2	0.0533 (9)	0.0253 (8)	0.0241 (7)	0.0066 (6)	0.0132 (6)	0.0012 (6)
O1	0.0845 (10)	0.0333 (7)	0.0306 (7)	0.0124 (6)	0.0255 (6)	0.0033 (5)
O2	0.0773 (10)	0.0365 (8)	0.0357 (8)	0.0039 (7)	0.0044 (7)	0.0003 (6)
O3	0.0656 (9)	0.0373 (8)	0.0401 (7)	0.0025 (6)	0.0037 (6)	0.0063 (6)

Geometric parameters (Å, º)

C10—O3	1.210 (2)	C6—O1	1.2293 (19)
C10—O2	1.320 (2)	C6—N2	1.345 (2)
C10—C11	1.494 (2)	C9—C8[i]	1.382 (2)
C11—C13	1.389 (2)	C9—H9	0.9300

C11—C12	1.392 (2)	C12—C13ii	1.382 (2)
C8—C9i	1.382 (2)	C12—H12	0.9300
C8—C7	1.389 (2)	C4—N2	1.410 (2)
C8—H8	0.9300	C13—C12ii	1.382 (2)
C7—C9	1.391 (2)	C13—H13	0.9300
C7—C6	1.494 (2)	C2—C1	1.374 (3)
C5—N1	1.331 (2)	C2—H2	0.9300
C5—C4	1.385 (2)	C1—N1	1.337 (2)
C5—H5	0.9300	C1—H1	0.9300
C3—C2	1.377 (2)	N2—H2N	0.89 (2)
C3—C4	1.387 (2)	O2—H1N	1.00 (3)
C3—H3	0.9300		
O3—C10—O2	123.83 (16)	C8i—C9—H9	119.6
O3—C10—C11	122.57 (16)	C7—C9—H9	119.6
O2—C10—C11	113.58 (15)	C13ii—C12—C11	119.97 (16)
C13—C11—C12	119.40 (16)	C13ii—C12—H12	120.0
C13—C11—C10	118.80 (15)	C11—C12—H12	120.0
C12—C11—C10	121.80 (16)	C5—C4—C3	118.38 (15)
C9i—C8—C7	120.14 (15)	C5—C4—N2	116.91 (15)
C9i—C8—H8	119.9	C3—C4—N2	124.68 (15)
C7—C8—H8	119.9	C12ii—C13—C11	120.63 (16)
C8—C7—C9	119.05 (15)	C12ii—C13—H13	119.7
C8—C7—C6	122.97 (14)	C11—C13—H13	119.7
C9—C7—C6	117.92 (14)	C1—C2—C3	119.90 (17)
N1—C5—C4	123.26 (16)	C1—C2—H2	120.1
N1—C5—H5	118.4	C3—C2—H2	120.1
C4—C5—H5	118.4	N1—C1—C2	122.36 (17)
C2—C3—C4	118.19 (16)	N1—C1—H1	118.8
C2—C3—H3	120.9	C2—C1—H1	118.8
C4—C3—H3	120.9	C5—N1—C1	117.91 (15)
O1—C6—N2	122.81 (16)	C6—N2—C4	126.60 (14)
O1—C6—C7	120.72 (14)	C6—N2—H2N	117.7 (12)
N2—C6—C7	116.47 (14)	C4—N2—H2N	115.4 (12)
C8i—C9—C7	120.81 (15)	C10—O2—H1N	111.7 (16)
O3—C10—C11—C13	9.3 (3)	N1—C5—C4—C3	−0.6 (3)
O2—C10—C11—C13	−169.05 (16)	N1—C5—C4—N2	177.29 (16)
O3—C10—C11—C12	−171.05 (17)	C2—C3—C4—C5	−0.4 (2)
O2—C10—C11—C12	10.6 (2)	C2—C3—C4—N2	−178.11 (16)
C9i—C8—C7—C9	0.6 (3)	C12—C11—C13—C12ii	−0.6 (3)
C9i—C8—C7—C6	177.70 (15)	C10—C11—C13—C12ii	179.14 (16)
C8—C7—C6—O1	−146.83 (17)	C4—C3—C2—C1	1.3 (3)
C9—C7—C6—O1	30.4 (2)	C3—C2—C1—N1	−1.3 (3)
C8—C7—C6—N2	33.8 (2)	C4—C5—N1—C1	0.6 (3)
C9—C7—C6—N2	−149.04 (16)	C2—C1—N1—C5	0.3 (3)
C8—C7—C9—C8i	−0.6 (3)	O1—C6—N2—C4	3.8 (3)
C6—C7—C9—C8i	−177.85 (15)	C7—C6—N2—C4	−176.86 (15)

C13—C11—C12—C13ii	0.6 (3)	C5—C4—N2—C6	157.60 (17)
C10—C11—C12—C13ii	−179.13 (16)	C3—C4—N2—C6	−24.7 (3)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*N*···O1iii	0.89 (2)	1.98 (2)	2.8616 (18)	171.3 (18)
O2—H1*N*···N1iv	1.00 (3)	1.69 (3)	2.6938 (19)	178 (2)

Symmetry codes: (iii) *x*, −*y*+1/2, *z*+1/2; (iv) *x*, *y*+1, *z*−1.