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

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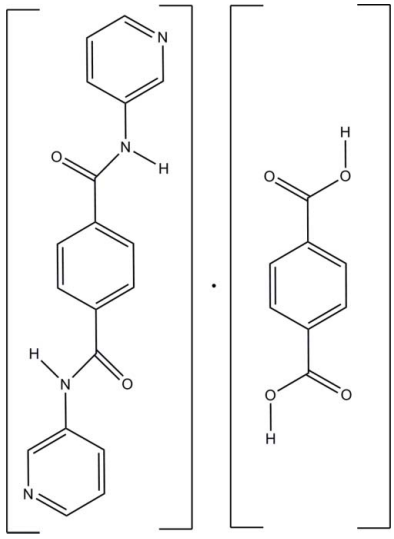
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.040; $wR$ factor = 0.095; data-to-parameter ratio = 11.3.

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 $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 296 K
0.25 × 0.24 × 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—H2*N*···O1$^{i}$ | 0.89 (2) | 1.98 (2) | 2.8616 (18) | 171.3 (18) |
| O2—H1*N*···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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Xiao, W., Xue, R. & Yin, Y. (2011). *Acta Cryst.* E**67**, o1333.

# supplementary materials

*Acta Cryst.* (2011). E**67**, o3064 [ **doi:10.1107/S1600536811041596** ]

# *N*,*N*'-Bis(pyridin-3-yl)terephthalamide-terephthalic acid (1/1)

**J. Lu, X. Liu, L. Li, Y. Chen and G. Shen**

## 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).

## 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.

## 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) = 1.2$U_{eq}$(C). H atoms bonded to O and N atoms were refined independently with isotropic displacement parameters.

## Figures

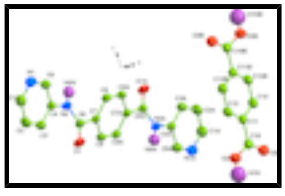

Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level (symmetry code; (A) -*x* + 1, -*y* + 1, -*z* + 1; (B) -*x* + 2, -*y* + 2, -*z* + 1). Hydrogen atoms bonded to C atoms are not shown.

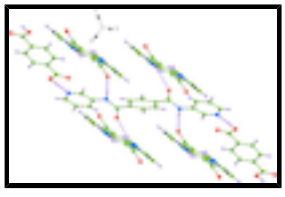

Fig. 2. Part of the crystal structure with hydrogen bonds shown as pink dashed lines. H atoms are purple.

### *N*,*N*'-Bis(pyridin-3-yl)terephthalamide; terephthalic acid

*Crystal data*

| | |
|---|---|
| $C_{18}H_{14}N_4O_2 \cdot C_8H_6O_4$ | $F(000) = 504$ |
| $M_r = 484.46$ | $D_x = 1.459$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 2279 reflections |
| $a = 11.0001$ (3) Å | $\theta = 2.7$–$25.2°$ |
| $b = 10.8080$ (2) Å | $\mu = 0.11$ mm$^{-1}$ |
| $c = 9.6903$ (2) Å | $T = 296$ K |
| $\beta = 106.830$ (2)° | Block, colourless |
| $V = 1102.73$ (4) Å$^3$ | 0.25 × 0.24 × 0.22 mm |
| $Z = 2$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD diffractometer | 1939 independent reflections |
| Radiation source: fine-focus sealed tube | 1640 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.035$ |
| φ and ω scans | $\theta_{max} = 25.0°$, $\theta_{min} = 1.9°$ |
| Absorption correction: multi-scan (SABADS; Sheldrick, 1996) | $h = -13 \rightarrow 12$ |
| $T_{min} = 0.974$, $T_{max} = 0.977$ | $k = -12 \rightarrow 11$ |
| 8117 measured reflections | $l = -11 \rightarrow 11$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.040$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.095$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.07$ | $w = 1/[\sigma^2(F_o^2) + (0.0378P)^2 + 0.3019P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 1939 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 171 parameters | $\Delta\rho_{max} = 0.14$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.19$ 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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, °)*

| | | | |
|---|---|---|---|
| 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—C13$^{ii}$ | 1.382 (2) |
| C8—C9$^{i}$ | 1.382 (2) | C12—H12 | 0.9300 |
| C8—C7 | 1.389 (2) | C4—N2 | 1.410 (2) |
| C8—H8 | 0.9300 | C13—C12$^{ii}$ | 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) | C8$^{i}$—C9—H9 | 119.6 |
| O3—C10—C11 | 122.57 (16) | C7—C9—H9 | 119.6 |
| O2—C10—C11 | 113.58 (15) | C13$^{ii}$—C12—C11 | 119.97 (16) |
| C13—C11—C12 | 119.40 (16) | C13$^{ii}$—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) |
| C9$^{i}$—C8—C7 | 120.14 (15) | C5—C4—N2 | 116.91 (15) |
| C9$^{i}$—C8—H8 | 119.9 | C3—C4—N2 | 124.68 (15) |
| C7—C8—H8 | 119.9 | C12$^{ii}$—C13—C11 | 120.63 (16) |
| C8—C7—C9 | 119.05 (15) | C12$^{ii}$—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) |
| C8$^{i}$—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) |
| C9$^{i}$—C8—C7—C9 | 0.6 (3) | C12—C11—C13—C12$^{ii}$ | −0.6 (3) |
| C9$^{i}$—C8—C7—C6 | 177.70 (15) | C10—C11—C13—C12$^{ii}$ | 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—C8$^{i}$ | −0.6 (3) | O1—C6—N2—C4 | 3.8 (3) |
| C6—C7—C9—C8$^{i}$ | −177.85 (15) | C7—C6—N2—C4 | −176.86 (15) |
| C13—C11—C12—C13$^{ii}$ | 0.6 (3) | C5—C4—N2—C6 | 157.60 (17) |
| C10—C11—C12—C13$^{ii}$ | −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—H2N···O1$^{iii}$ | 0.89 (2) | 1.98 (2) | 2.8616 (18) | 171.3 (18) |
| O2—H1N···N1$^{iv}$ | 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.

**Fig. 1**

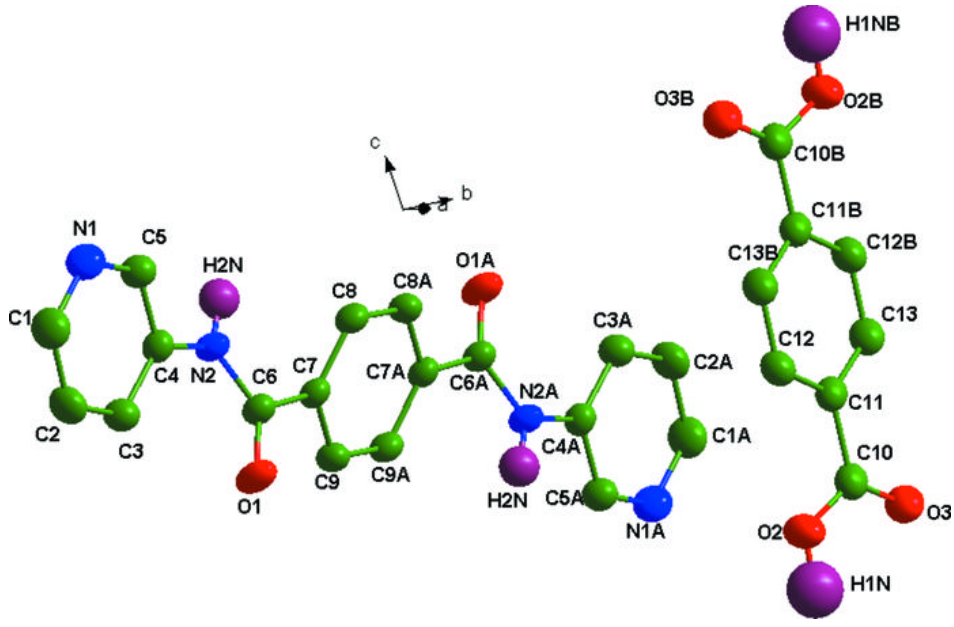
H1NB
O3B
O2B
C10B
C11B
C13B
C12B
C13
C12
C11
C10
O3
O2
H1N
c
b
a
N1
C5
H2N
C1
C4
N2
C6
C7
C8
C8A
O1A
C7A
C6A
N2A
C3A
C2A
C1A
C4A
C5A
N1A
H2N
C2
C3
O1
C9
C9A

**Fig. 2**