

2-Aminoterephthalic acid–4,4'-bipyridine (1/1)

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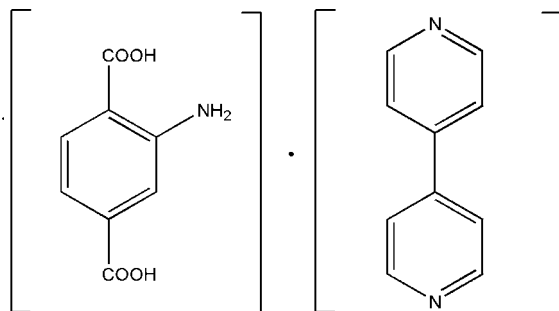
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; disorder in main residue; R factor = 0.067; wR factor = 0.199; data-to-parameter ratio = 11.6.

The asymmetric unit of the title compound, $\text{C}_{10}\text{H}_8\text{N}_2 \cdot \text{C}_8\text{H}_7\text{NO}_4$, contains two half-molecules, which constitute a 1:1 co-crystal. The 2-aminoterephthalic acid molecule is situated on an inversion center being disordered between two orientations in a 1:1 ratio. In the 4,4'-bipyridine molecule, which is situated on a twofold rotational axis, the two pyridine rings form a dihedral angle of $37.5(1)^\circ$. In the crystal, molecules are held together *via* intermolecular $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonds. The crystal packing exhibits $\pi-\pi$ interactions between the aromatic rings with a centroid-centroid distance of $3.722(3)$ Å.

Related literature

For the crystal structures of polymeric coordination polymers with 2-aminoterephthalic acid linkers, see: Ma *et al.* (2005); Bauer *et al.* (2008).



Experimental

Crystal data

$\text{C}_{10}\text{H}_8\text{N}_2 \cdot \text{C}_8\text{H}_7\text{NO}_4$
 $M_r = 337.33$
 Monoclinic, $C2/c$
 $a = 16.9501(18)$ Å
 $b = 11.1959(13)$ Å
 $c = 9.251(1)$ Å
 $\beta = 116.986(2)^\circ$

$V = 1564.4(3)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 293$ K
 $0.33 \times 0.19 \times 0.11$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.967$, $T_{\max} = 0.989$

3865 measured reflections
 1370 independent reflections
 894 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.075$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.199$
 $S = 1.00$
 1370 reflections

118 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.32$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.29$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N1}-\text{H1A} \cdots \text{O2}^{\text{i}}$	0.86	2.08	2.931 (5)	172
$\text{N1}-\text{H1B} \cdots \text{O2}^{\text{ii}}$	0.86	1.95	2.619 (5)	133
$\text{O1}-\text{H1} \cdots \text{N2}^{\text{iii}}$	0.82	1.82	2.635 (3)	170

Symmetry codes: (i) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + \frac{3}{2}, -y + \frac{3}{2}, -z$; (iii) $-x + 1, y, -z + \frac{3}{2}$.

Data collection: SMART (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); 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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5085).

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supplementary materials

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2-Aminoterephthalic acid-4,4'-bipyridine (1/1)

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Comment

Bipyridine is a well known molecule often used as a linker in polymeric coordination compounds. 2-Aminoterephthalic acid is also sometimes used as a linker in polymeric compounds (Ma *et al.*, 2005; Bauer *et al.*, 2008). The title compound (I) is a 1:1 cocrystal of the aforementioned linkers. Herewith we present its crystal structure.

In (I) (Fig. 1), the 2-aminoterephthalic acid molecule is situated on an inversion center, therefore, it is disordered (namely, the amino group is disordered between two positions). The carboxyl groups are twisted from the benzene ring plane at $11.4 (1)^\circ$. The 4,4'-bipyridine molecule is situated on a twofold rotational axis, and two pyridine rings form a dihedral angle of $37.5 (1)^\circ$.

In the crystal structure, the molecules are held together *via* intermolecular N—H \cdots O and O—H \cdots N hydrogen bonds. The crystal packing exhibits π — π interactions between the aromatic rings with the centroid-to-centroid distance of $3.722 (3) \text{ \AA}$.

Experimental

2-Diaminoterephthalic acid(10 mmol) and 4,4'-bipyridine(10 mmol) were dissolved to methanol (10 ml) and then hydrochloric acid(5 ml) was added. A few minutes later, an methanol solution (10 ml) of tin tetrachloride (5 mmol) was added with stirring. The reaction mixture was stirred for 4 h. The solution was held at room temperature for about two weeks, whereupon yellow crystals of the title compound, which were beyond expectations, were obtained.

Refinement

All H-atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 \AA , N—H = 0.86 \AA , O—H = 0.82 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$, $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Figures

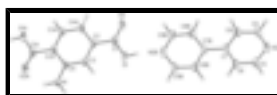


Fig. 1. The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. (symmetry code A: $x + 3/2, -y + 3/2, -z$ and B: $-x + 1, y, -z + 3/2$). For the disordered molecule of 2-aminoterephthalic acid, only one orientation is shown.

2-Aminobenzene-1,4-dicarboxylic acid-4,4'-bipyridine (1/1)

Crystal data

$\text{C}_{10}\text{H}_8\text{N}_2 \cdot \text{C}_8\text{H}_7\text{NO}_4$

$M_r = 337.33$

Monoclinic, $C2/c$

$F(000) = 704$

$D_x = 1.432 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

supplementary materials

$a = 16.9501 (18) \text{ \AA}$	Cell parameters from 1178 reflections
$b = 11.1959 (13) \text{ \AA}$	$\theta = 2.3\text{--}26.0^\circ$
$c = 9.251 (1) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$\beta = 116.986 (2)^\circ$	$T = 293 \text{ K}$
$V = 1564.4 (3) \text{ \AA}^3$	Block, yellow
$Z = 4$	$0.33 \times 0.19 \times 0.11 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer	1370 independent reflections
Radiation source: fine-focus sealed tube graphite	894 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.075$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 2.3^\circ$
$T_{\text{min}} = 0.967$, $T_{\text{max}} = 0.989$	$h = -20 \rightarrow 12$
3865 measured reflections	$k = -11 \rightarrow 13$
	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.067$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.199$	H-atom parameters constrained
$S = 1.00$	$w = 1/[\sigma^2(F_o^2) + (0.1295P)^2]$
1370 reflections	where $P = (F_o^2 + 2F_c^2)/3$
118 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$

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 (\AA^2)

x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
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N1	0.8270 (3)	0.9576 (4)	-0.0096 (6)	0.0607 (13)	0.50
H1A	0.8317	1.0134	0.0577	0.073*	0.50
H1B	0.8478	0.9678	-0.0779	0.073*	0.50
N2	0.39951 (15)	0.8071 (2)	1.0092 (3)	0.0600 (7)	
O1	0.67530 (15)	0.82272 (19)	0.2964 (3)	0.0742 (8)	
H1	0.6550	0.8097	0.3602	0.111*	
O2	0.65519 (15)	0.62839 (19)	0.2565 (3)	0.0711 (7)	
C1	0.68006 (18)	0.7229 (3)	0.2283 (3)	0.0520 (8)	
C2	0.71594 (15)	0.7367 (2)	0.1106 (3)	0.0436 (7)	
C3	0.75521 (17)	0.8419 (2)	0.1014 (3)	0.0459 (7)	
H3	0.7595	0.9041	0.1713	0.055*	
C4	0.78845 (17)	0.8581 (2)	-0.0081 (3)	0.0462 (7)	
H4	0.8134	0.9307	-0.0140	0.055*	0.50
C5	0.39774 (19)	0.9000 (3)	0.9217 (3)	0.0594 (8)	
H5	0.3689	0.9685	0.9295	0.071*	
C6	0.43583 (18)	0.9020 (2)	0.8196 (3)	0.0529 (8)	
H6	0.4328	0.9707	0.7606	0.063*	
C7	0.47874 (16)	0.8020 (2)	0.8042 (3)	0.0451 (7)	
C8	0.47940 (19)	0.7037 (3)	0.8939 (3)	0.0564 (8)	
H8	0.5062	0.6331	0.8861	0.068*	
C9	0.4404 (2)	0.7104 (3)	0.9947 (4)	0.0646 (9)	
H9	0.4427	0.6436	1.0563	0.077*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.094 (4)	0.048 (2)	0.071 (3)	-0.013 (2)	0.064 (3)	-0.013 (2)
N2	0.0592 (15)	0.0888 (17)	0.0500 (14)	-0.0024 (13)	0.0404 (13)	0.0035 (12)
O1	0.1089 (18)	0.0821 (14)	0.0749 (15)	0.0006 (12)	0.0797 (15)	-0.0074 (11)
O2	0.0983 (17)	0.0765 (14)	0.0719 (15)	0.0073 (12)	0.0679 (14)	0.0144 (11)
C1	0.0551 (16)	0.0688 (19)	0.0402 (15)	0.0119 (14)	0.0287 (13)	0.0078 (13)
C2	0.0456 (14)	0.0573 (15)	0.0366 (14)	0.0069 (12)	0.0264 (12)	0.0046 (11)
C3	0.0504 (15)	0.0575 (15)	0.0381 (14)	0.0071 (12)	0.0273 (13)	-0.0029 (11)
C4	0.0520 (16)	0.0550 (15)	0.0440 (15)	0.0023 (12)	0.0324 (13)	0.0046 (11)
C5	0.0643 (18)	0.0808 (19)	0.0509 (18)	0.0050 (15)	0.0416 (16)	-0.0027 (14)
C6	0.0640 (17)	0.0658 (17)	0.0448 (15)	0.0036 (14)	0.0385 (14)	0.0031 (12)
C7	0.0429 (14)	0.0644 (16)	0.0342 (13)	-0.0031 (12)	0.0229 (12)	-0.0010 (11)
C8	0.0624 (17)	0.0661 (17)	0.0552 (17)	0.0057 (14)	0.0394 (15)	0.0089 (13)
C9	0.073 (2)	0.0802 (19)	0.0576 (18)	-0.0005 (16)	0.0443 (17)	0.0180 (15)

Geometric parameters (\AA , $^\circ$)

N1—C4	1.295 (5)	C3—H3	0.9300
N1—H1A	0.8600	C4—C2 ⁱ	1.403 (4)
N1—H1B	0.8600	C4—H4	0.9300
N2—C5	1.310 (3)	C5—C6	1.365 (3)
N2—C9	1.325 (4)	C5—H5	0.9300
O1—C1	1.304 (3)	C6—C7	1.378 (3)

supplementary materials

O1—H1	0.8200	C6—H6	0.9300
O2—C1	1.210 (3)	C7—C8	1.376 (3)
C1—C2	1.476 (4)	C7—C7 ⁱⁱ	1.477 (5)
C2—C3	1.374 (4)	C8—C9	1.368 (4)
C2—C4 ⁱ	1.403 (4)	C8—H8	0.9300
C3—C4	1.376 (4)	C9—H9	0.9300
C4—N1—H1A	120.0	C3—C4—H4	120.5
C4—N1—H1B	120.0	C2 ⁱ —C4—H4	120.5
H1A—N1—H1B	120.0	N2—C5—C6	123.8 (3)
C5—N2—C9	116.9 (2)	N2—C5—H5	118.1
C1—O1—H1	109.5	C6—C5—H5	118.1
O2—C1—O1	122.8 (3)	C5—C6—C7	119.7 (3)
O2—C1—C2	123.5 (3)	C5—C6—H6	120.1
O1—C1—C2	113.7 (3)	C7—C6—H6	120.1
C3—C2—C4 ⁱ	119.0 (2)	C8—C7—C6	116.6 (2)
C3—C2—C1	120.6 (2)	C8—C7—C7 ⁱⁱ	122.47 (18)
C4 ⁱ —C2—C1	120.4 (2)	C6—C7—C7 ⁱⁱ	120.88 (17)
C2—C3—C4	122.0 (2)	C9—C8—C7	119.5 (3)
C2—C3—H3	119.0	C9—C8—H8	120.2
C4—C3—H3	119.0	C7—C8—H8	120.2
N1—C4—C3	119.8 (3)	N2—C9—C8	123.4 (3)
N1—C4—C2 ⁱ	121.1 (3)	N2—C9—H9	118.3
C3—C4—C2 ⁱ	119.0 (2)	C8—C9—H9	118.3
N1—C4—H4	3.0		

Symmetry codes: (i) $-x+3/2, -y+3/2, -z$; (ii) $-x+1, y, -z+3/2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots O2 ⁱⁱⁱ	0.86	2.08	2.931 (5)	172
N1—H1B \cdots O2 ⁱ	0.86	1.95	2.619 (5)	133
O1—H1 \cdots N2 ⁱⁱ	0.82	1.82	2.635 (3)	170

Symmetry codes: (iii) $-x+3/2, y+1/2, -z+1/2$; (i) $-x+3/2, -y+3/2, -z$; (ii) $-x+1, y, -z+3/2$.

Fig. 1

