

Di-2-pyridyl disulfide–succinic acid (1/1)

Cheng Zhang, Ling Yuan, Ji-Yong Liu and Wei Xu*

State Key Laboratory Base of Novel Functional Materials and Preparation Science, Faculty of Materials Science and Chemical Engineering, Institute of Solid Materials Chemistry, Ningbo University, Ningbo, Zhejiang 315211, People's Republic of China

Correspondence e-mail: zhengyueqing@nbu.edu.cn

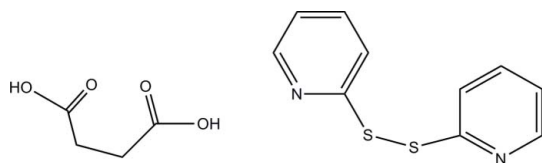
Received 19 March 2009; accepted 30 April 2009

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.053; wR factor = 0.139; data-to-parameter ratio = 14.1.

In the title compound, $\text{C}_{10}\text{H}_8\text{N}_2\text{S}_2 \cdot \text{C}_4\text{H}_6\text{O}_4$, both components of the cocrystal lie on crystallographic twofold rotation axes. In the di-2-pyridyl disulfide molecule, the dihedral angle between the two pyridine rings is $66.6(1)^\circ$. In the crystal structure, intermolecular $\text{O}-\text{H} \cdots \text{N}$ and weak $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds link both types of molecules into columns along the c axis.

Related literature

For general background to the design of cocrystals, see: Desiraju (2003); Thalladi *et al.* (2007). For a related structure, see: Raghavan *et al.* (1977).



Experimental

Crystal data

$\text{C}_{10}\text{H}_8\text{N}_2\text{S}_2 \cdot \text{C}_4\text{H}_6\text{O}_4$
 $M_r = 338.39$

Monoclinic, $C2/c$
 $a = 8.4211(17)$ Å
 $b = 13.347(3)$ Å
 $c = 14.141(3)$ Å
 $\beta = 98.43(3)^\circ$

$V = 1572.2(6)$ Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.36$ mm⁻¹
 $T = 293$ K
 $0.60 \times 0.47 \times 0.23$ mm

Data collection

Rigaku R-AXIS RAPID
 diffractometer
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.822$, $T_{\max} = 0.921$

7089 measured reflections
 1799 independent reflections
 1490 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.139$
 $S = 1.28$
 1799 reflections
 128 parameters

H atoms treated by a mixture of
 independent and constrained
 refinement
 $\Delta\rho_{\max} = 0.40$ e Å⁻³
 $\Delta\rho_{\min} = -0.30$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O2}-\text{H2H} \cdots \text{N1}$	0.83 (4)	1.94 (4)	2.759 (3)	173 (4)
$\text{C2}-\text{H2A} \cdots \text{O1}^i$	0.93	2.47	3.128 (4)	127

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

This project was sponsored by the K. C. Wong Magna Fund of Ningbo University and supported by the Expert Project of Key Basic Research of the Ministry of Science and Technology of China (grant No. 2003CCA00800), the Zhejiang Provincial Natural Science Foundation (grant No. Z203067) and the Ningbo Municipal Natural Science Foundation (grant No. 2006A610061).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2792).

References

- Desiraju, G. R. (2003). *J. Mol. Struct.* **656**, 5-15.
 Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Raghavan, N. V. & Seff, K. (1977). *Acta Cryst.* **B33**, 386-391.
 Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
 Rigaku/MS (2004). *CrystalStructure*. Rigaku/MS, The Woodlands, Texas, USA.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112-122.
 Thalladi, V. R., Dabros, M., Gehrke, A., Weiss, H. C. & Boese, R. (2007). *Cryst. Growth Des.* **7**, 598-599.

supplementary materials

Acta Cryst. (2009). E65, o1213 [doi:10.1107/S1600536809016213]

Di-2-pyridyl disulfide-succinic acid (1/1)

C. Zhang, L. Yuan, J.-Y. Liu and W. Xu

Comment

The design of cocrystals has been a field of intensive research in recent years. With reliable design strategies, cocrystals could offer a modular approach to developing materials with desirable properties. (Desiraju, 2003; Thalladi *et al.*, 2007). Weak noncovalent interactions such as hydrogen bonds are utilized to create cocrystals. Herein we report the structure of the title cocrystal.

The formula unit of the title compound (Fig. 1) contains one molecule of di-2-pyridyl disulfide (dpds) and one molecule succinic acid. The dihedral angle between the two pyridine rings of the dpds molecule is $66.6(1)^\circ$, and the S—S bond length, $2.025(2) \text{ \AA}$, is not significantly different than that found in the structure of the free ligand, $2.016(2) \text{ \AA}$ (Raghavan *et al.*, 1977). The torsion angle of the C6-C7-C7ⁱⁱ-C6ⁱⁱ [symmetry code: (ii) $-x, y, -z+3/2$] backbone of succinic acid is $74.5(3)^\circ$. The proton of the carboxylate O atom (O2) of the succinic acid molecule forms a strong hydrogen bond with atom N1 of the dpds molecule (see Table 1 for hydrogen bond geometry). In addition, in the crystal structure, weak intermolecular C-H \cdots O hydrogen bonds supplement intermolecular N-H \cdots O hydrogen bonds to form columns running parallel to the *c*-axis (Fig 2).

Experimental

All chemicals were reagent grade quality obtained from commercial sources and without further purification. Dpds (0.2206 g, 1 mmol) and succinic acid (0.1181 g, 1 mmol) were dissolved in a H₂O/EtOH solution ($v/v = 2:1$, 15 ml), which was stirred for 0.5 h and then filtrated, the filtrate was allowed to concentrate by slow evaporation to give colorless block crystals.

Refinement

H atoms bonded to C atoms were placed in geometrically calculated positions (C-H = $0.93\text{--}0.97 \text{ \AA}$) and were refined in a riding-model approximation, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$. The H atom bonded to O2 atoms was located in a difference Fourier map and its position refined with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$.

Figures

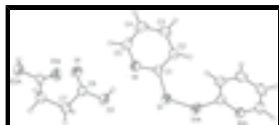


Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 45% probability level. The complete molecules of di-2-pyridyl disulfide and succinic acid are generated by the symmetry operators $(-x, y, -z+1/2)$ and $(-x, y, -z+3/2)$ respectively.

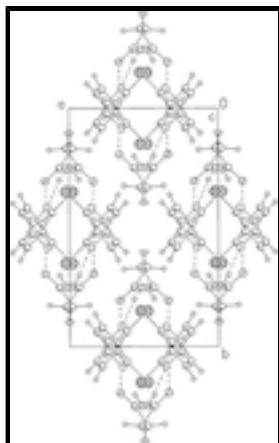


Fig. 2. Part of the crystal structure of the title compound with hydrogen bonds shown as dashed lines.

Di-2-pyridyl disulfide-succinic acid (1/1)

Crystal data

$C_{10}H_8N_2S_2 \cdot C_4H_6O_4$

$M_r = 338.39$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 8.4211 (17) \text{ \AA}$

$b = 13.347 (3) \text{ \AA}$

$c = 14.141 (3) \text{ \AA}$

$\beta = 98.43 (3)^\circ$

$V = 1572.2 (6) \text{ \AA}^3$

$Z = 4$

$F_{000} = 704$

$D_x = 1.430 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4565 reflections

$\theta = 3.1\text{--}27.5^\circ$

$\mu = 0.36 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, colorless

$0.60 \times 0.47 \times 0.23 \text{ mm}$

Data collection

Rigaku R-Axis RAPID
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 0 pixels mm^{-1}

$T = 293 \text{ K}$

ω scans

Absorption correction: multi-scan
(ABSCOR; Higashi, 1995)

$T_{\min} = 0.822$, $T_{\max} = 0.921$

7089 measured reflections

1799 independent reflections

1490 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 27.5^\circ$

$\theta_{\min} = 3.1^\circ$

$h = -10 \rightarrow 10$

$k = -17 \rightarrow 17$

$l = -18 \rightarrow 16$

Refinement

Refinement on F^2

Secondary atom site location: difference Fourier map

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.053$$

$$wR(F^2) = 0.139$$

$$S = 1.28$$

1799 reflections

128 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0282P)^2 + 3.2667P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.40 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	-0.1474 (3)	0.58448 (17)	0.44834 (16)	0.0396 (6)
C1	-0.1175 (3)	0.56306 (19)	0.36047 (19)	0.0349 (6)
C2	-0.1815 (4)	0.4813 (2)	0.3079 (2)	0.0436 (7)
H2A	-0.1580	0.4692	0.2466	0.052*
C3	-0.2816 (4)	0.4183 (2)	0.3497 (3)	0.0518 (8)
H3A	-0.3273	0.3627	0.3165	0.062*
C4	-0.3132 (4)	0.4382 (2)	0.4408 (3)	0.0540 (8)
H4A	-0.3792	0.3961	0.4702	0.065*
C5	-0.2449 (4)	0.5218 (2)	0.4873 (2)	0.0474 (7)
H5A	-0.2673	0.5356	0.5484	0.057*
S1	0.02142 (10)	0.65005 (6)	0.32235 (6)	0.0473 (3)
C6	-0.0647 (3)	0.7501 (2)	0.63801 (19)	0.0362 (6)
C7	0.0187 (4)	0.8324 (2)	0.69918 (19)	0.0395 (6)
H7A	-0.0128	0.8965	0.6699	0.047*
H7B	0.1337	0.8254	0.7008	0.047*
O1	-0.1622 (3)	0.69379 (16)	0.66296 (15)	0.0513 (6)
O2	-0.0193 (3)	0.74777 (17)	0.55229 (15)	0.0523 (6)
H2H	-0.065 (5)	0.701 (3)	0.521 (3)	0.079*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0424 (14)	0.0434 (13)	0.0345 (12)	0.0021 (11)	0.0106 (10)	0.0002 (9)
C1	0.0338 (15)	0.0335 (13)	0.0394 (14)	0.0023 (11)	0.0122 (11)	0.0003 (10)
C2	0.0500 (19)	0.0368 (15)	0.0474 (17)	-0.0022 (13)	0.0184 (13)	-0.0075 (12)
C3	0.055 (2)	0.0339 (15)	0.069 (2)	-0.0067 (14)	0.0180 (16)	-0.0032 (14)
C4	0.053 (2)	0.0447 (17)	0.069 (2)	-0.0020 (15)	0.0237 (16)	0.0166 (15)
C5	0.0502 (19)	0.0556 (18)	0.0399 (16)	0.0046 (15)	0.0177 (13)	0.0109 (13)
S1	0.0518 (5)	0.0440 (4)	0.0515 (5)	-0.0135 (4)	0.0255 (4)	-0.0117 (3)
C6	0.0394 (16)	0.0363 (14)	0.0330 (13)	0.0031 (12)	0.0057 (11)	0.0053 (10)
C7	0.0424 (16)	0.0387 (14)	0.0372 (15)	-0.0043 (12)	0.0054 (12)	0.0025 (11)
O1	0.0617 (15)	0.0510 (13)	0.0432 (12)	-0.0169 (11)	0.0140 (10)	0.0003 (9)
O2	0.0669 (16)	0.0549 (14)	0.0390 (12)	-0.0156 (12)	0.0205 (10)	-0.0064 (9)

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

N1—C1	1.334 (3)	C5—H5A	0.9300
N1—C5	1.345 (4)	S1—S1 ⁱ	2.0251 (17)
C1—C2	1.385 (4)	C6—O1	1.204 (3)
C1—S1	1.787 (3)	C6—O2	1.324 (3)
C2—C3	1.384 (4)	C6—C7	1.507 (4)
C2—H2A	0.9300	C7—C7 ⁱⁱ	1.516 (5)
C3—C4	1.380 (5)	C7—H7A	0.9700
C3—H3A	0.9300	C7—H7B	0.9700
C4—C5	1.378 (5)	O2—H2H	0.83 (4)
C4—H4A	0.9300		
C1—N1—C5	117.2 (3)	N1—C5—H5A	118.5
N1—C1—C2	123.9 (3)	C4—C5—H5A	118.5
N1—C1—S1	111.3 (2)	C1—S1—S1 ⁱ	106.10 (10)
C2—C1—S1	124.8 (2)	O1—C6—O2	123.7 (3)
C3—C2—C1	117.6 (3)	O1—C6—C7	124.6 (3)
C3—C2—H2A	121.2	O2—C6—C7	111.7 (2)
C1—C2—H2A	121.2	C6—C7—C7 ⁱⁱ	113.6 (2)
C4—C3—C2	119.7 (3)	C6—C7—H7A	108.8
C4—C3—H3A	120.2	C7 ⁱⁱ —C7—H7A	108.8
C2—C3—H3A	120.2	C6—C7—H7B	108.8
C5—C4—C3	118.5 (3)	C7 ⁱⁱ —C7—H7B	108.8
C5—C4—H4A	120.7	H7A—C7—H7B	107.7
C3—C4—H4A	120.7	C6—O2—H2H	109 (3)
N1—C5—C4	123.1 (3)		

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2H \cdots N1	0.83 (4)	1.94 (4)	2.759 (3)	173 (4)

C2—H2A \cdots O1ⁱⁱⁱ

0.93

2.47

3.128 (4)

127

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

Fig. 1

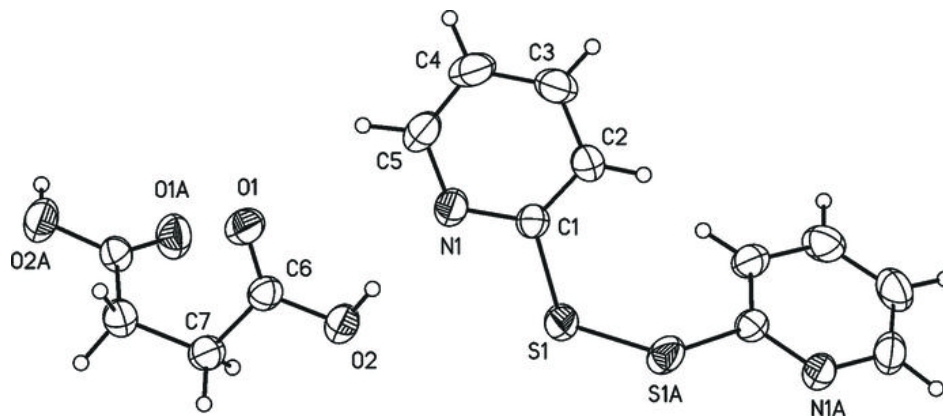


Fig. 2

