organic compounds

Bis(4-pyridyl) disulfide–2,2′-[(p-phenyl-enebis(oxy)]diacetic acid (1/1)

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The asymmetric unit of the title 1:1 co-crystal, $C_{10}H_8N_2S_2 \cdot C_{10}H_{10}O_6$, comprises two half-molecules, the bis(4-pyridyl) disulfide having twofold rotational symmetry and the 2,2′-[(p-phenylenebis(oxy)]diacetic acid having crystallographic inversion symmetry. In the disulfide molecule, the dihedral angle between the two pyridine rings is 86.8 (1)°, while the carboxyl groups of the substituted quinone lie essentially in the plane of the benzene ring [dihedral angle = 5.3 (1)°]. In the crystal, the components are linked via intermolecular O—H⋯N hydrogen bonds into zigzag chains which extend along c and are interlinked through C—H⋯π associations.

Related literature

For the use of bis(4-pyridyl)disulfide (bpds) as a linker in the construction of coordination polymers, see: Kondo et al. (2000); Zhu et al. (2010).
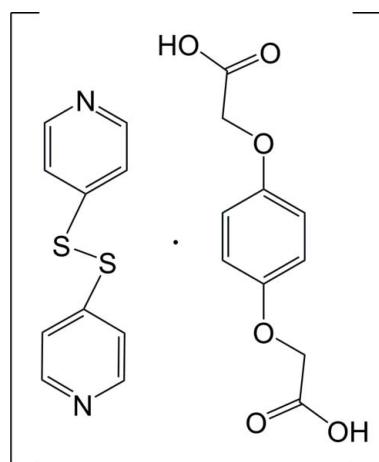
Experimental

Crystal data

$C_{10}H_8N_2S_2 \cdot C_{10}H_{10}O_6$	V = 2029.4 (5) Å^3
M_r = 446.50	Z = 4
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
a = 14.331 (1) Å	μ = 0.30 mm^{-1}
b = 5.057 (1) Å	T = 296 K
c = 28.003 (3) Å	$0.31 \times 0.21 \times 0.09$ mm
β = 90.200 (5)°	

Data collection

Bruker APEXII CCD area-detector diffractometer	4893 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2001)	1761 independent reflections
T_{min} = 0.912, T_{max} = 0.974	1450 reflections with $I > 2\sigma(I)$
	R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043	137 parameters
$wR(F^2)$ = 0.114	H-atom parameters constrained
S = 1.05	$\Delta\rho_{max}$ = 0.62 e Å^{-3}
1761 reflections	$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

$Cg1$ is the centroid of the C8–C10/C8′–C10′ ring.

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O3—H3⋯N1^i	0.82	1.81	2.629 (3)	174
C7—H7B⋯$Cg1^{ii}$	0.97	2.76	3.528 (2)	136

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

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SIR97 (Altomare et al., 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: WinGX (Farrugia, 1999).

References

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supporting information

Bis(4-pyridyl) disulfide–2,2′-[(*p*-phenylenebis(oxy)]diacetic acid (1/1)

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S1. Comment

Bis(4-pyridyl)disulfide (bpds) is often used as a linker in the construction of coordination polymers because of its flexibility (Kondo *et al.*, 2000; Zhu *et al.*, 2010). The attempt at synthesizing a Cd^{II} coordination polymer using bis(4-pyridyl)disulfide and hydroquinone-*O,O′*-diacetic acid (H$_2$qda) as ligands gave instead the 1:1 title co-crystal C$_{10}$H$_8$N$_2$S$_2$. C$_{10}$H$_{10}$O$_6$, and the crystal structure is reported here.

In the title compound, the asymmetric unit comprises two half molecules, the bis(4-pyridyl)disulfide having twofold rotational symmetry and the hydroquinone-*O,O′*-diacetic acid having crystallographic inversion symmetry (Fig. 1). In the disulfide molecule, the dihedral angle between the two pyridine rings is 93.2 (1)° while the carboxylic acid groups of the substituted quinone molecule lie essentially in the plane of the benzene ring [dihedral angle, 5.3 (1)°]. In the crystal, the two components are linked *via* intermolecular O—H⋯N hydrogen bonds into one-dimensional zigzag chains which extend along *c* (Fig. 2) and are inter-linked through C—H⋯π associations (Table 1, Fig. 3).

S2. Experimental

A mixture of hydroquinone-*O,O′*-diacetic acid (H$_2$qda) (0.023 g, 0.1 mmol), bis(4-pyridyl)disulfide (bpds) (0.022 g, 0.1 mmol) and Cd(NO$_3$)$_2$. 4H$_2$O (0.038 g, 0.1 mmol) in H$_2$O (7.0 ml) was placed in a 16 ml Teflon-lined stainless steel vessel and heated to 160 °C for 48 h, then cooled to room temperature at a rate of -5 °C/h. The solution was filtered and the colorless filtrate was allowed to stand at room temperature. Slow evaporation for about one week afforded colorless block crystals.

S3. Refinement

All H atoms bonded to C atoms were added according to theoretical models, assigned isotropic displacement parameters and allowed to ride on their respective parent atoms [C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C)]. The carboxylic acid H atom was located from the Fourier map and allowed to ride on the parent O atom in the final cycles of refinement, with the O—H distance being fixed at 0.82 Å with U_{iso}(H) = 1.5U_{eq}(O).


Figure 1
Atom numbering scheme and anisotropic displacement ellipsoid plot of (I) at the 50% probability level. H atoms are represented by circles of arbitrary size. Symmetry codes: (i) -*x* + 2, *y*, -*z* + 1/2; (ii) -*x*, -*y* + 2, -*z*.
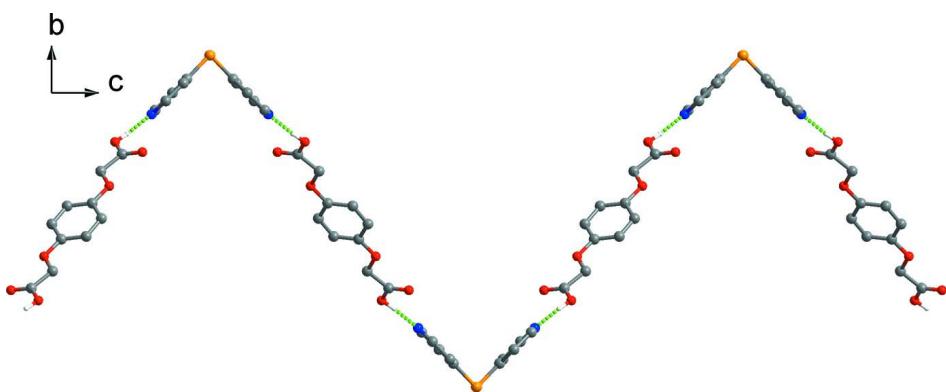
Figure 2

The one-dimensional zigzag chain structure of the title compound. Non-associative H atoms are omitted and hydrogen bonds are shown as dashed lines.
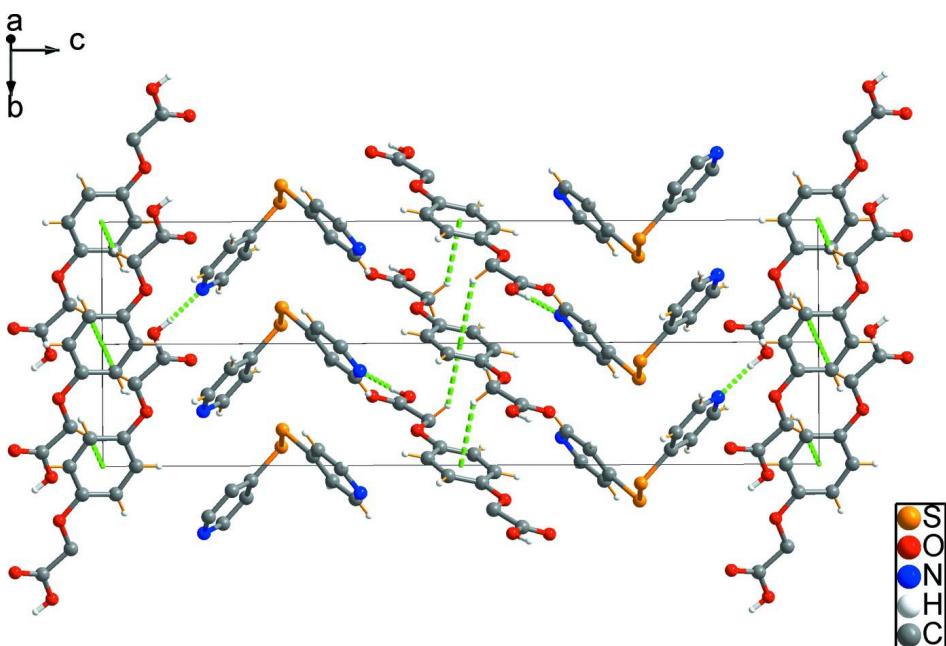
Figure 3

The packing diagram of the title compound showing C—H···π interactions.

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Crystal data
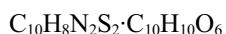
$C_{10}H_8N_2S_2$·$C_{10}H_{10}O_6$	$F(000) = 928$
$M_r = 446.50$	$D_x = 1.461$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 1634 reflections
$a = 14.331$ (1) Å	$\theta = 2.8–25.2°$
$b = 5.057$ (1) Å	$\mu = 0.30$ mm^{-1}
$c = 28.003$ (3) Å	$T = 296$ K
$\beta = 90.200$ (5)°	Block, colorless
$V = 2029.4$ (5) Å^3	$0.31 \times 0.21 \times 0.09$ mm
$Z = 4$	

Data collection

Bruker APEXII CCD area-detector diffractometer	4893 measured reflections
	1761 independent reflections
Radiation source: fine-focus sealed tube	1450 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.026$
φ and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 1.5°$
Absorption correction: multi-scan	$h = -10 \rightarrow 16$
(*SADABS*; Bruker, 2001)	$k = -5 \rightarrow 5$
$T_{min} = 0.912$, $T_{max} = 0.974$	$l = -33 \rightarrow 32$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.043$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.114$	
$S = 1.05$	H-atom parameters constrained
1761 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 1.8236P]$
137 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.62$ e Å^{-3}
	$\Delta\rho_{min} = -0.29$ 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 (Å²)

	x	y	z	$U_{iso}*/U_{eq}$
S1	0.92972 (4)	−0.22476 (13)	0.24909 (2)	0.0514 (2)
O1	0.07968 (11)	0.6344 (3)	0.06109 (5)	0.0485 (4)
O2	0.15067 (12)	0.2818 (4)	0.12321 (6)	0.0596 (5)
N1	0.83733 (14)	0.3882 (5)	0.14055 (7)	0.0543 (6)
O3	0.26328 (12)	0.1757 (4)	0.07197 (7)	0.0683 (6)
H3	0.2843	0.0923	0.0947	0.102*
C8	0.04234 (15)	0.8125 (4)	0.02905 (8)	0.0389 (5)
C9	−0.02803 (15)	0.9711 (5)	0.04649 (8)	0.0440 (6)
H9	−0.0471	0.9519	0.0780	0.053*
C7	0.15542 (15)	0.4802 (5)	0.04525 (8)	0.0465 (6)
H7A	0.2061	0.5945	0.0353	0.056*
H7B	0.1364	0.3737	0.0181	0.056*
C3	0.89942 (16)	0.0216 (4)	0.20664 (8)	0.0425 (5)
C6	0.18774 (15)	0.3037 (5)	0.08524 (9)	0.0462 (6)
C1	0.92798 (17)	0.3451 (5)	0.14718 (8)	0.0517 (6)
H1	0.9703	0.4408	0.1289	0.062*

C2	0.96216 (16)	0.1654 (5)	0.17981 (8)	0.0476 (6)
H2	1.0261	0.1414	0.1837	0.057*
C10	−0.07061 (15)	1.1580 (5)	0.01785 (8)	0.0443 (6)
H10	−0.1181	1.2636	0.0300	0.053*
C4	0.80519 (17)	0.0660 (6)	0.19985 (10)	0.0584 (7)
H4	0.7612	−0.0283	0.2173	0.070*
C5	0.77771 (19)	0.2495 (6)	0.16724 (11)	0.0639 (8)
H5	0.7142	0.2800	0.1633	0.077*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0554 (4)	0.0495 (4)	0.0492 (4)	−0.0077 (3)	−0.0039 (3)	0.0034 (3)
O1	0.0518 (10)	0.0482 (10)	0.0455 (9)	0.0114 (8)	0.0044 (7)	0.0065 (8)
O2	0.0509 (10)	0.0769 (13)	0.0511 (10)	0.0127 (9)	0.0051 (8)	0.0143 (9)
N1	0.0542 (13)	0.0617 (14)	0.0470 (11)	0.0169 (11)	−0.0012 (9)	0.0002 (10)
O3	0.0555 (11)	0.0901 (15)	0.0593 (11)	0.0314 (10)	0.0075 (9)	0.0199 (10)
C8	0.0398 (12)	0.0344 (12)	0.0426 (12)	−0.0008 (9)	−0.0022 (9)	0.0013 (9)
C9	0.0490 (13)	0.0446 (14)	0.0384 (12)	0.0034 (11)	0.0044 (10)	0.0003 (10)
C7	0.0410 (12)	0.0475 (15)	0.0508 (13)	0.0035 (10)	0.0000 (10)	0.0048 (11)
C3	0.0492 (13)	0.0423 (13)	0.0361 (11)	0.0021 (10)	−0.0012 (9)	−0.0069 (10)
C6	0.0397 (13)	0.0484 (14)	0.0504 (14)	−0.0009 (11)	−0.0047 (11)	0.0023 (11)
C1	0.0531 (15)	0.0570 (16)	0.0451 (13)	0.0090 (12)	0.0057 (11)	0.0037 (12)
C2	0.0424 (13)	0.0553 (15)	0.0452 (13)	0.0083 (11)	0.0005 (10)	0.0001 (11)
C10	0.0426 (12)	0.0420 (13)	0.0481 (13)	0.0054 (10)	0.0044 (10)	−0.0011 (10)
C4	0.0454 (14)	0.0656 (18)	0.0643 (16)	−0.0013 (13)	0.0037 (12)	0.0045 (14)
C5	0.0468 (15)	0.075 (2)	0.0701 (18)	0.0106 (14)	−0.0039 (13)	0.0013 (16)

Geometric parameters (Å, º)

S1—C3	1.775 (2)	C7—C6	1.504 (3)
S1—S1^i	2.0150 (14)	C7—H7A	0.9700
O1—C8	1.378 (3)	C7—H7B	0.9700
O1—C7	1.409 (3)	C3—C2	1.381 (3)
O2—C6	1.195 (3)	C3—C4	1.381 (3)
N1—C1	1.330 (3)	C1—C2	1.378 (3)
N1—C5	1.336 (4)	C1—H1	0.9300
O3—C6	1.316 (3)	C2—H2	0.9300
O3—H3	0.8200	C10—C8^{ii}	1.384 (3)
C8—C9	1.379 (3)	C10—H10	0.9300
C8—C10^{ii}	1.384 (3)	C4—C5	1.359 (4)
C9—C10	1.380 (3)	C4—H4	0.9300
C9—H9	0.9300	C5—H5	0.9300
C3—S1—S1^i	105.02 (8)	O2—C6—O3	125.0 (2)
C8—O1—C7	117.02 (17)	O2—C6—C7	125.5 (2)
C1—N1—C5	117.5 (2)	O3—C6—C7	109.5 (2)
C6—O3—H3	109.5	N1—C1—C2	123.1 (2)

C9—C8—O1	115.61 (19)	N1—C1—H1	118.5
C9—C8—C10ⁱⁱ	119.4 (2)	C2—C1—H1	118.5
O1—C8—C10ⁱⁱ	125.0 (2)	C1—C2—C3	118.5 (2)
C8—C9—C10	121.0 (2)	C1—C2—H2	120.7
C8—C9—H9	119.5	C3—C2—H2	120.7
C10—C9—H9	119.5	C9—C10—C8ⁱⁱ	119.7 (2)
O1—C7—C6	109.23 (19)	C9—C10—H10	120.2
O1—C7—H7A	109.8	C8ⁱⁱ—C10—H10	120.2
C6—C7—H7A	109.8	C5—C4—C3	119.0 (2)
O1—C7—H7B	109.8	C5—C4—H4	120.5
C6—C7—H7B	109.8	C3—C4—H4	120.5
H7A—C7—H7B	108.3	N1—C5—C4	123.4 (2)
C2—C3—C4	118.5 (2)	N1—C5—H5	118.3
C2—C3—S1	125.16 (18)	C4—C5—H5	118.3
C4—C3—S1	116.30 (18)		
C7—O1—C8—C9	−176.4 (2)	C5—N1—C1—C2	0.3 (4)
C7—O1—C8—C10ⁱⁱ	3.2 (3)	N1—C1—C2—C3	0.5 (4)
O1—C8—C9—C10	179.7 (2)	C4—C3—C2—C1	−0.5 (3)
C10ⁱⁱ—C8—C9—C10	0.1 (4)	S1—C3—C2—C1	178.12 (18)
C8—O1—C7—C6	178.70 (18)	C8—C9—C10—C8ⁱⁱ	−0.1 (4)
S1ⁱ—S1—C3—C2	3.2 (2)	C2—C3—C4—C5	−0.2 (4)
S1ⁱ—S1—C3—C4	−178.13 (17)	S1—C3—C4—C5	−179.0 (2)
O1—C7—C6—O2	5.5 (3)	C1—N1—C5—C4	−1.1 (4)
O1—C7—C6—O3	−174.7 (2)	C3—C4—C5—N1	1.1 (4)

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

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C8–C10/C8′–C10′ ring.

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O3—H3⋯N1ⁱⁱⁱ	0.82	1.81	2.629 (3)	174
C7—H7*B*⋯Cg1ⁱᵛ	0.97	2.76	3.528 (2)	136

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