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A novel three-dimensional supramolecule of 2,2′-dithiodibenzoic acid–4,4′-bipyridine (1/1)

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Key indicators

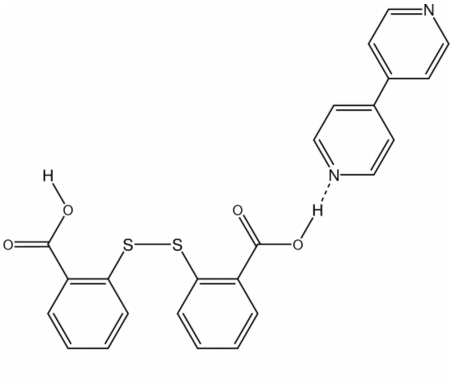
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.046
wR factor = 0.121
Data-to-parameter ratio = 12.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title complex, $C_{14}H_{10}O_4S_2 \cdot C_{10}H_8N_2$, hydrogen bonds between the carboxyl O atoms and the N atoms of 4,4′-bipyridine link the two different molecules. There is a weak interaction between the carboxyl O atom and an H atom of 4,4′-bipyridine, contributing to the formation of a three-dimensional supramolecule.

Comment

Supramolecules are not just collections of molecules; their characteristic properties are distinct from the aggregate properties of the constituent molecular species (Reddy *et al.*, 1995). In the opinion of structural chemists, a crystal of an organic compound is the perfect supramolecule. At a supramolecular level, the main target in crystal engineering is to synthesize intriguing structures, such as ribbons, sheets, layers, tubes, channels and cavities, which might be appropriately functionalized to obtain materials that act as templates for catalysts or organic zeolites (Desiraju, 1995).



(I)

In this paper, we report a new co-crystal, (I), of 2,2′-dithiodibenzoic acid with 4,4′-bipyridine. 4,4′-Bipyridine is an excellent synthon in preparing novel structures, owing to its rigidity and aptness to form strong hydrogen bonds *via* its two N atoms. Many supramolecular crystals of 4,4′-bipyridine have been reported (Lough *et al.*, 2000). Except for typical hydrogen bonds (Chatterjee *et al.*, 1998), weak interactions between carboxyl O atoms and the H atoms of pyridyl are often found in these structures. In (I), the dihedral angle between the two pyridyl rings of 4,4′-bipyridine is 29.7 (8)°. The distance between the C8–C13 plane and the N2-pyridyl ring is less than 3.5 Å, and the dihedral angle between the two planes is only 9.0 (2)°. The distance between O2 and N1 is 2.601 (5) Å, indicating a strong hydrogen bond (Fig. 1 and Table 2). The two C=O bonds, *viz.* C1—O1 and C14—O4, are

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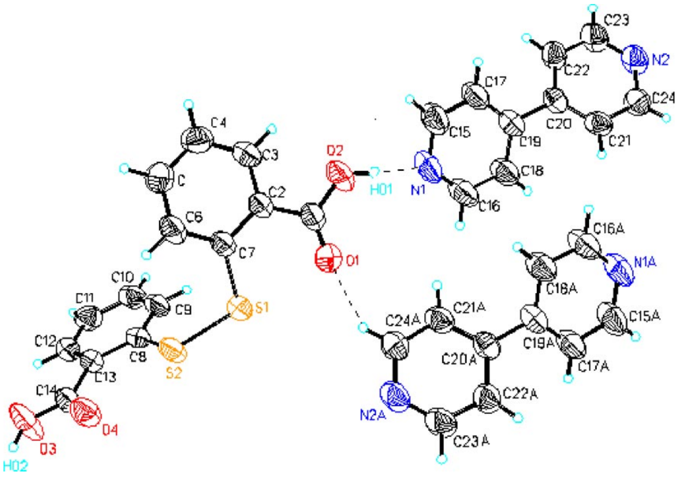

Figure 1
The molecular structure of (I). **[Can an ellipsoid plot be provided with labelled C atoms; if so, please also provide the ellipsoid probability]**

1.201 (3) and 1.205 (3) Å, respectively; these are much shorter than the other C—O bonds (Table 1). The O1···H24*A* distance is 2.46 Å, which is typical for a weak interaction. The two kinds of weak interactions in the structure allow the two molecules to form an extended three-dimensional supramolecule (Fig. 2.).

Experimental

2,2′-Dithiodibenzoic acid (0.0153 g, 0.5 mmol) was dissolved in 15 ml methanol, and a solution of 4,4′-bipyridine (0.0047 g, 0.3 mmol) in 5 ml methanol was added dropwise under intense agitation. The mixture was placed in air at room temperature. After about a week, colorless crystals of (I) were obtained in 60% yield.

Crystal data

$C_{14}H_{10}O_4S_2 \cdot C_{10}H_8N_2$
$M_r = 462.52$
Monoclinic, $P2_1/c$
$a = 9.2079$ (4) Å
$b = 13.2553$ (5) Å
$c = 17.8928$ (8) Å
$\beta = 93.385$ (1)°
$V = 2180.07$ (16) Å^3
$Z = 4$
$D_x = 1.409$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 120 reflections
$\theta = 1.9$–25.0°
$\mu = 0.28$ mm^{-1}
$T = 293$ (2) K
Prism, colorless
0.42 × 0.20 × 0.14 mm

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.693$, $T_{\max} = 0.962$
7140 measured reflections
3830 independent reflections
2973 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.021$
$\theta_{\max} = 25.0°$
$h = -10 \rightarrow 6$
$k = -15 \rightarrow 9$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.121$
$S = 1.07$
3830 reflections
297 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 1.4907P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.010$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

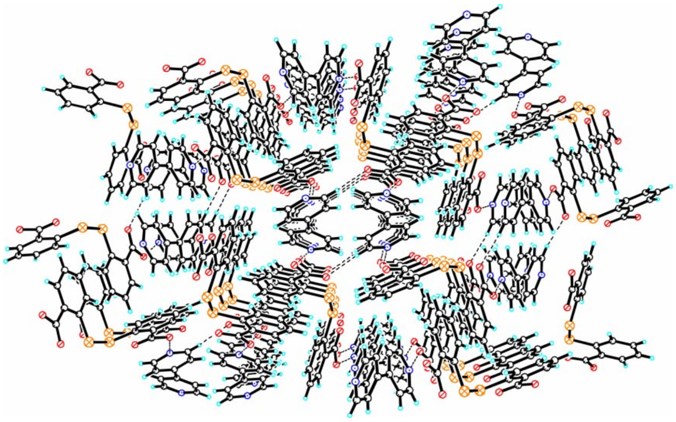

Figure 2
The three-dimensional packing structure of (I), viewed along the *a* aixs.

Table 1
Selected geometric parameters (Å, °).

S1—C7	1.794 (3)	C1—O2	1.315 (3)
S1—S2	2.0572 (10)	C14—O4	1.205 (3)
S2—C8	1.795 (3)	C14—O3	1.310 (3)
C1—O1	1.201 (3)		
C7—S1—S2	104.59 (9)	O1—C1—O2	123.2 (3)
C8—S2—S1	104.65 (9)	O4—C14—O3	123.4 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—HO1···N1$^{\rm i}$	0.86 (5)	1.86 (3)	2.644 (2)	151 (3)
C24—H24*A*···O1$^{\rm ii}$	0.93	2.46	3.188 (3)	135
O3—HO2···N2$^{\rm iii}$	0.88 (5)	1.75 (3)	2.618 (5)	170 (5)
C4—H4*A*···O4$^{\rm iv}$	0.93	2.67	3.404 (1)	136

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

The positions of the H atoms bonded to carbon were generated geometrically (C—H bond lengths fixed at 0.96 Å), and allowed to ride on their respective parent atoms. The positional parameters of H atoms bonded to oxygen were refined with isotropic displacement parameters.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); program(s) used to solve structure: *SHELXTL* (Siemens, 1994);; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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