Crystal structure of 4,4'-((6-(dimethylamino)-1,3,5-triazine-2,4-diyl)-bis(sulfanediyl))dibenzoic acid, $C_{19}H_{16}N_4O_4S_2$

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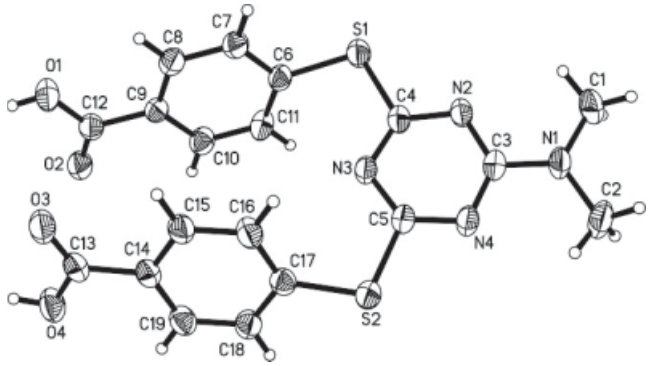

Abstract

$C_{19}H_{16}N_4O_4S_2$, monoclinic, *C*2/*c* (no. 15), $a = 21.523(6)$ Å, $b = 9.531(3)$ Å, $c = 19.115(5)$ Å, $\beta = 92.700(4)°$, $V = 3916.8$ Å^3, $Z = 8$, $R_{gt(F)} = 0.0426$, $wR_{ref(F^2)} = 0.1152$, $T = 296$ K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.18×0.23×0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.07 cm^{-1}
Diffractometer, scan mode:	Bruker APEX2 CCD, φ and ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10723, 3857
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2381
$N(param)_{refined}$:	264
Programs:	SAINT, SADABS [6] SIR97 [7], SHELX [8], WinGX [9]

Source of material

A mixture of 4,4'-((6-(dimethylamino)-1,3,5-triazine-2,4-diyl)-bis(sulfanediyl))dibenzoic acid (0.43 g, 0.1 mmol), bis(4-pyridyl)disulfide (*bpds*) (0.022 g, 0.1 mmol) and $Cd(NO_3)_2{\cdot}4H_2O$ (0.038 g, 0.1 mmol) in H_2O (7.0 ml) was placed in a 23 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 colourless filtrate was allowed to evapora.te. After one week colourless block-shaped crystals were obtained.

Experimental details

All H atoms bonded to C atoms were added according to theoretical models and allowed to ride on their respective parent atoms with C–H = 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C)$. The H atom attached to O were located from the Fourier map and allowed to ride on their parent oxygen atoms in the final cycles of refinement, with the O–H distance being fixed at 0.82 Å and $U_{iso}(H) = 1.5U_{eq}(O)$.

Discussion

The design and synthesis of metal-organic frameworks (MOFs) with well-regulated architectures have caused increasing attention due to their potential applications as functional materials [1, 2]. Flexible ligands have been paid much attention during the self-assembly process. Flexible ligands may adopt several kinds of conformations when coordinate to metal ions, which make it more difficult to predict and control the final coordination networks. So the use of flexible ligands in the formation of coordination polymers may generate novel complexes with interesting topologies and attractive properties [3-5]. The title compound was obtained unintentionally during attempts to synthesize a Cd^{II} coordination polymer using the title compound as ligand. The molecluar structure of the title compound is shown in the figure. In the title structure, the dihedral angle between the benzene rings of the two 4-mercaptobenzoic acid groups and the triazine ring are 48.5° and 61.5°, respectively. The molecule and its symmetry equivalent are connected via O–H···O hydrogen bonds to dimers.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*f*	0.4377	0.8750	0.1987	0.104
H(4)	8*f*	0.5424	1.1250	0.3335	0.114
H(15)	8*f*	0.4101	0.8503	0.4280	0.071
H(7)	8*f*	0.2332	0.7004	0.3374	0.072
H(18)	8*f*	0.3795	1.2762	0.5350	0.075
H(19)	8*f*	0.4553	1.2538	0.4546	0.076
H(8)	8*f*	0.3230	0.7327	0.2776	0.071
H(10)	8*f*	0.3098	1.1469	0.3045	0.073
H(11)	8*f*	0.2190	1.1160	0.3620	0.075
H(16)	8*f*	0.3348	0.8714	0.5088	0.073
H(1B)	8*f*	0.0189	0.8903	0.5675	0.124
H(1A)	8*f*	–0.0091	1.0240	0.6020	0.124
H(1C)	8*f*	0.0008	0.8863	0.6460	0.124
H(2A)	8*f*	0.0726	0.9802	0.7354	0.129
H(2B)	8*f*	0.0683	1.1337	0.7061	0.129
H(2C)	8*f*	0.1329	1.0576	0.7132	0.129

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(2)	8*f*	0.29723(3)	1.09727(8)	0.59791(4)	0.0555(4)	0.0788(5)	0.0504(4)	–0.0022(3)	0.0078(3)	–0.0070(4)
S(1)	8*f*	0.14550(3)	0.87924(8)	0.39612(4)	0.0530(4)	0.0916(5)	0.0512(4)	–0.0103(4)	0.0101(3)	–0.0070(4)
N(4)	8*f*	0.1821(1)	1.0371(2)	0.6113(1)	0.053(1)	0.072(1)	0.046(1)	0.005(1)	0.014(1)	0.000(1)
N(2)	8*f*	0.11382(9)	0.9390(2)	0.5218(1)	0.049(1)	0.066(1)	0.053(1)	–0.003(1)	0.015(1)	0.002(1)
N(1)	8*f*	0.0796(1)	0.9901(2)	0.6319(1)	0.054(1)	0.077(2)	0.058(1)	0.003(1)	0.022(1)	–0.001(1)
O(1)	8*f*	0.40811(8)	0.8536(2)	0.2221(1)	0.056(1)	0.077(1)	0.077(1)	0.006(1)	0.022(1)	0.000(1)
N(3)	8*f*	0.21945(9)	0.9872(2)	0.4987(1)	0.046(1)	0.063(1)	0.044(1)	0.002(1)	0.0106(9)	0.001(1)
C(14)	8*f*	0.4400(1)	1.0499(3)	0.4327(1)	0.041(1)	0.061(2)	0.054(2)	0.001(1)	0.003(1)	0.006(1)
C(3)	8*f*	0.1262(1)	0.9891(3)	0.5872(1)	0.054(2)	0.057(2)	0.050(2)	0.008(1)	0.015(1)	0.006(1)
O(4)	8*f*	0.51696(9)	1.1442(2)	0.3627(1)	0.065(1)	0.076(1)	0.090(2)	–0.009(1)	0.031(1)	0.004(1)
O(3)	8*f*	0.49925(9)	0.9141(2)	0.3568(1)	0.060(1)	0.071(1)	0.090(2)	–0.002(1)	0.026(1)	–0.005(1)
O(2)	8*f*	0.39976(9)	1.0865(2)	0.2320(1)	0.070(1)	0.068(1)	0.082(2)	–0.011(1)	0.027(1)	–0.009(1)
C(5)	8*f*	0.2253(1)	1.0328(2)	0.5643(1)	0.050(1)	0.052(1)	0.045(2)	0.008(1)	0.009(1)	0.006(1)
C(13)	8*f*	0.4883(1)	1.0306(3)	0.3805(1)	0.040(1)	0.068(2)	0.062(2)	–0.000(1)	0.003(1)	0.006(2)
C(9)	8*f*	0.3250(1)	0.9427(3)	0.2846(1)	0.050(2)	0.059(2)	0.041(1)	–0.002(1)	0.005(1)	–0.004(1)
C(17)	8*f*	0.3492(1)	1.0765(3)	0.5299(1)	0.045(1)	0.060(2)	0.052(2)	0.003(1)	–0.000(1)	0.001(1)
C(15)	8*f*	0.4039(1)	0.9364(3)	0.4496(2)	0.049(2)	0.057(2)	0.074(2)	0.002(1)	0.010(1)	–0.006(1)
C(12)	8*f*	0.3813(1)	0.9675(3)	0.2440(1)	0.049(2)	0.070(2)	0.049(2)	0.001(1)	0.001(1)	–0.003(1)
C(7)	8*f*	0.2483(1)	0.7905(3)	0.3304(1)	0.066(2)	0.055(2)	0.059(2)	–0.007(1)	0.014(1)	–0.003(1)
C(6)	8*f*	0.2171(1)	0.9048(3)	0.3553(1)	0.053(2)	0.066(2)	0.038(1)	–0.001(1)	0.006(1)	–0.001(1)
C(18)	8*f*	0.3856(1)	1.1902(3)	0.5134(1)	0.063(2)	0.057(2)	0.069(2)	–0.003(1)	0.014(1)	–0.006(2)
C(4)	8*f*	0.1620(1)	0.9427(2)	0.4812(1)	0.050(2)	0.053(2)	0.050(2)	0.002(1)	0.013(1)	0.004(1)
C(19)	8*f*	0.4309(1)	1.1769(3)	0.4653(2)	0.057(2)	0.058(2)	0.075(2)	–0.008(1)	0.013(1)	0.004(2)
C(8)	8*f*	0.3022(1)	0.8100(3)	0.2947(1)	0.062(2)	0.057(2)	0.059(2)	0.002(1)	0.012(1)	–0.007(1)
C(10)	8*f*	0.2941(1)	1.0571(3)	0.3105(1)	0.071(2)	0.057(2)	0.056(2)	–0.002(1)	0.016(1)	0.003(1)
C(11)	8*f*	0.2401(1)	1.0386(3)	0.3453(1)	0.072(2)	0.060(2)	0.058(2)	0.011(1)	0.022(1)	0.003(1)
C(16)	8*f*	0.3588(1)	0.9487(3)	0.4978(1)	0.053(2)	0.057(2)	0.074(2)	–0.003(1)	0.016(1)	0.003(2)
C(1)	8*f*	0.0172(1)	0.9437(3)	0.6100(2)	0.054(2)	0.101(2)	0.096(3)	–0.004(2)	0.030(2)	–0.004(2)
C(2)	8*f*	0.0892(1)	1.0451(4)	0.7027(2)	0.084(2)	0.114(3)	0.062(2)	0.011(2)	0.031(2)	–0.010(2)

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