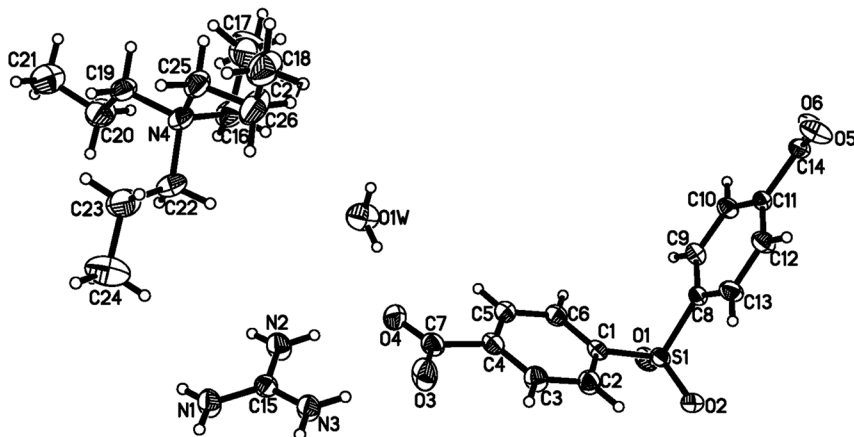


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Crystal structure of tetrapropylammonium guanidinium 4,4'-sulfonyldibenzoate monohydrate, $C_{27}H_{44}N_4O_7S$



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Abstract

$C_{27}H_{44}N_4O_7S$, monoclinic, $P2_1/n$ (no. 14), $a = 10.216(3)$ Å, $b = 18.133(5)$ Å, $c = 17.036(5)$ Å, $\beta = 98.371(3)^\circ$, $V = 3122.2(16)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0435$, $wR_{ref}(F^2) = 0.1333$, $T = 296(2)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

4,4'-Sulfonyldibenzoic acid (A. R.), guanidine hydrochloride (A. R.) and tetrapropylammonium hydroxide (25% aqueous solution) were mixed in a molar ratio of 10:10:1. The mixture was dissolved in a minimum amount of ethanol and water to form the clean solution, then the solution was stirred for about 0.5 h. Subsequently the clean solution was set aside to

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.40 × 0.30 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.15 mm ⁻¹
Diffractometer, scan mode:	BRUKER APEX-II, φ and ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	20,315, 5496, 0.025
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4660
$N(param)_{refined}$:	352
Programs:	BRUKER [1], SHELX [2, 3]

allow it slow evaporation at room temperature. Colorless block crystals were obtained about seven days later.

Comment

4,4'-Sulfonyldibenzoic acid, as a V-shape molecule with flexible benzene rings, can take part in the formation of various hydrogen bonds after the deprotonation of the terminal carboxyl groups. Searching in the CSD database [4], there exist hundreds of MOFs constructed with 4,4'-sulfonyldibenzoic acid. It can be concluded that this compound can display varied spatial configurations and has strong abilities to form hydrogen bonds. However, the pure organic crystal structures of 4,4'-sulfonyldibenzoic acid are seldom reported compared with the related MOFs [5, 6]. Thus, the crystal structure of the title compound will enrich the corresponding organic crystal structures.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.29582 (16)	0.12453 (9)	0.95133 (11)	0.0394 (4)
N1	−0.35892 (16)	0.09333 (10)	0.49290 (10)	0.0594 (5)
H1A	−0.4300	0.1191	0.4911	0.071*
H1B	−0.3545	0.0586	0.4590	0.071*
O1	0.41940 (13)	0.04032 (7)	1.06220 (8)	0.0544 (4)
S1	0.38869 (4)	0.11686 (2)	1.04727 (3)	0.04099 (15)
C2	0.18696 (18)	0.17094 (11)	0.94029 (12)	0.0498 (5)
H2A	0.1657	0.1997	0.9818	0.060*
N2	−0.14583 (16)	0.06837 (10)	0.54895 (11)	0.0606 (5)
H2B	−0.0781	0.0780	0.5837	0.073*
H2C	−0.1419	0.0337	0.5149	0.073*
O2	0.31935 (13)	0.15680 (8)	1.10117 (8)	0.0543 (4)
C3	0.11055 (19)	0.17365 (11)	0.86634 (12)	0.0520 (5)
H3A	0.0374	0.2047	0.8583	0.062*
N3	−0.26318 (16)	0.16009 (9)	0.59952 (10)	0.0529 (4)
H3B	−0.3343	0.1858	0.5977	0.063*
H3C	−0.1965	0.1691	0.6352	0.063*
O3	−0.04629 (15)	0.17454 (10)	0.71916 (9)	0.0734 (5)
C4	0.14098 (17)	0.13096 (10)	0.80408 (11)	0.0444 (4)
N4	0.08536 (18)	0.15126 (10)	0.27330 (9)	0.0553 (4)
O4	0.07160 (14)	0.08491 (9)	0.67381 (8)	0.0615 (4)
C5	0.25260 (17)	0.08600 (11)	0.81627 (11)	0.0464 (4)
H5A	0.2753	0.0581	0.7745	0.056*
O5	0.88805 (13)	0.35269 (8)	1.02285 (11)	0.0665 (4)
C6	0.33000 (17)	0.08245 (10)	0.88976 (11)	0.0457 (4)
H6A	0.4042	0.0521	0.8977	0.055*
O6	1.00180 (12)	0.25136 (8)	1.06179 (9)	0.0600 (4)
C7	0.04827 (19)	0.13080 (12)	0.72564 (12)	0.0519 (5)
O1W	0.25168 (18)	0.03266 (9)	0.58227 (12)	0.0818 (5)
H1WA	0.2052	0.0470	0.6166	0.123*
H1WB	0.2946	0.0723	0.5642	0.123*
C8	0.53784 (16)	0.16473 (9)	1.04204 (10)	0.0378 (4)
C9	0.65150 (17)	0.12744 (10)	1.03027 (11)	0.0427 (4)
H9A	0.6501	0.0767	1.0224	0.051*
C10	0.76715 (16)	0.16707 (10)	1.03040 (11)	0.0436 (4)
H10A	0.8447	0.1424	1.0240	0.052*
C11	0.76919 (16)	0.24311 (10)	1.04004 (10)	0.0383 (4)
C12	0.65278 (18)	0.27931 (11)	1.04854 (13)	0.0510 (5)
H12A	0.6524	0.3304	1.0528	0.061*
C13	0.53724 (18)	0.24042 (11)	1.05075 (13)	0.0510 (5)
H13A	0.4599	0.2649	1.0580	0.061*
C14	0.89718 (17)	0.28569 (10)	1.04169 (11)	0.0429 (4)
C15	−0.25641 (17)	0.10712 (10)	0.54782 (11)	0.0444 (4)
C16	0.1906 (3)	0.11511 (15)	0.33308 (14)	0.0744 (7)
H16A	0.1640	0.0646	0.3406	0.089*
H16B	0.1926	0.1403	0.3835	0.089*
C17	0.3304 (3)	0.1143 (2)	0.31212 (19)	0.0955 (10)
H17A	0.3267	0.1234	0.2557	0.115*
H17B	0.3811	0.1538	0.3404	0.115*
C18	0.3975 (4)	0.0446 (2)	0.3320 (4)	0.1507 (19)
H18A	0.4850	0.0466	0.3178	0.226*
H18B	0.4031	0.0358	0.3880	0.226*
H18C	0.3488	0.0053	0.3034	0.226*
C19	0.0783 (2)	0.11491 (11)	0.19247 (12)	0.0570 (5)
H19A	0.0099	0.1396	0.1564	0.068*

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H19B	0.1617	0.1235	0.1731	0.068*
C20	0.0507 (3)	0.03360 (14)	0.18826 (16)	0.0810 (8)
H20A	−0.0353	0.0239	0.2038	0.097*
H20B	0.1168	0.0077	0.2247	0.097*
C21	0.0530 (3)	0.00610 (15)	0.10529 (19)	0.0919 (9)
H21A	0.0353	−0.0459	0.1031	0.138*
H21B	−0.0132	0.0314	0.0694	0.138*
H21C	0.1386	0.0152	0.0903	0.138*
C22	−0.0447 (2)	0.14257 (14)	0.30508 (14)	0.0674 (6)
H22A	−0.0344	0.1628	0.3584	0.081*
H22B	−0.0630	0.0903	0.3090	0.081*
C23	−0.1638 (3)	0.17903 (19)	0.25638 (18)	0.0880 (8)
H23A	−0.1564	0.2322	0.2620	0.106*
H23B	−0.1654	0.1670	0.2008	0.106*
C24	−0.2910 (3)	0.1534 (2)	0.2833 (3)	0.1161 (12)
H24A	−0.3648	0.1771	0.2518	0.174*
H24B	−0.2990	0.1010	0.2770	0.174*
H24C	−0.2900	0.1661	0.3381	0.174*
C25	0.1177 (2)	0.23175 (12)	0.26025 (12)	0.0615 (6)
H25A	0.2025	0.2339	0.2411	0.074*
H25B	0.0519	0.2511	0.2186	0.074*
C26	0.1238 (4)	0.28223 (15)	0.33148 (15)	0.0921 (9)
H26A	0.0409	0.2798	0.3528	0.111*
H26B	0.1941	0.2663	0.3725	0.111*
C27	0.1491 (4)	0.36003 (16)	0.30769 (18)	0.0967 (10)
H27A	0.1528	0.3917	0.3531	0.145*
H27B	0.2317	0.3623	0.2872	0.145*
H27C	0.0789	0.3758	0.2675	0.145*

In the asymmetric unit of the title compound, the contents are one 4,4'-sulfonyldibenzoate, one guanidinium cation, one tetrapropylammonium cation and one water molecule. In the anion, the dihedral angle between two benzene rings is 107.1°, and two terminal carboxyl groups are distorted 4.5° and 24.5° from their related benzene rings. Obviously, the anion displays the V-shape skeleton and two terminal carboxyl groups are not coplanar with their mother rings [7]. From the crystal packing model, it can be seen that 4,4'-sulfonyldibenzoate can link with guanidine cation by N–H...O contacts, and the linkage motif can be further connected with the water molecules by N–H...O and O–H...O hydrogen bonds to generate a three-dimensional host lattice with regular channels. It is noticeable that the weak hydrogen bonds of C–H...S and C–H...O are also present to further consolidate the crystal.

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