# Crystal structure of 2,2′-(1,4-butanediyl)bis(3*H*-benzimidazolium) sulfate heptahydrate, $[C_{18}H_{20}N_4][SO_4] \cdot 7H_2O$

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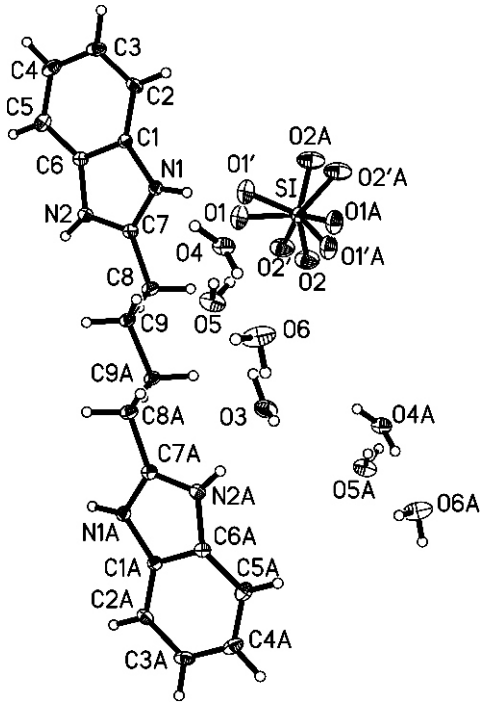


**Abstract**

$C_{18}H_{34}N_4O_{11}S$, monoclinic, *C*12/*c*1 (no. 15), $a$ = 11.168(4) Å, $b$ = 13.022(4) Å, $c$ = 17.743(6) Å, $\beta$ = 100.899(4)°, $V$ = 2533.8 Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.068, $wR_{ref}(F^2)$ = 0.160, $T$ = 143 K.

**Source of material**

2,2′-(1,4-butanediyl)bis(3*H*-benzimidazole) (87.0 mg, 0.3 mmol) was put in 14 mL (0.05 M) of sulfuric acid aqueous solution, and then was sealed in a 25 mL Teflon-lined stainless steel container, which was heated at 423 K for 72 h. Some colourless block-shaped crystals were obtained after cooled down to room temperature at a rate of 5 K/h and isolated for X-ray diffraction measurement.

**Experimental details**

All hydrogen atoms bound to carbon atoms were placed in calculated positions and refined in a riding model with $d$(C—H) = 0.95 Å (C$sp^2$) or 0.99 Å (C$sp^3$), and $U_{iso}$(H) = 1.2 $U_{eq}$(C). Hydrogen atoms bound to nitrogen and oxygen atoms were found in difference Fourier maps and refined freely. The oxygen atoms of sulfate are disordered over two sites with occupancies of 0.88(1):0.12(1). The disorder of spherical group $SO_4^{2-}$ in the crystal structure is a primary reason for the relatively large $R$ values. The H atoms bound to N1, N2 and O4 were obtained by difference Fourier syntheses. The other H atoms are set in theoretical positions. No additional solvent molecule was found.

**Discussion**

Organic ligands, especially the bridging ligands containing oxygen and nitrogen have attracted a considerable interest in recent years because of coordination polymers. Bis(2-benzimidazoles) and some substituted bis(2-benzimidazolyl)alkanes are alternative choices because of their multifunctional linking groups [1]. They are known to be strong chelating agents coordinating through both nitrogen atoms [2]. Many complexes based on them have been reported because of their wide-ranging antivirus activity [3], importance in selective ion-exchange resins [4] and mimicking biological systems [5].

The crystal structure of the title compound shows two protons from the one sulfuric acid molecule transferred to the N atoms of two benzimidazoles, respectively. There are six annular structures encircling the S atom in the crystal structure. These annular structures formed mostly by hydrogen bonds between sulfate anion and water molecules. Each sulfate anion is linked by these rings, and forms a reticular inorganic layer in (001). On the other hand, 2,2′-(1,4-butanediyl)bis(3*H*-benzimidazolium) is fixed in the inorganic layer by hydrogen bonds with $d$(N1–H1N···O1) = 2.67 Å, ∠N1–H1N···O1 = 165.8°; $d$(N2A–H2N···O3) = 2.75 Å, and ∠N2A–H2N···O3 = 168.3°, respectively. The dihedral angle of N1/C1/C6/N2/C7 ring with C1/C2/C3/C4/C5/C6 ring is 1.9(2)°, indicating such rings being almost coplanar. The $\pi$-$\pi$ stacking interactions of the aromatic rings link the organic cations into an organic layer, which is also parallel (001). Organic and inorganic layers are interlacing in the crystal structure.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | colorless block, size 0.17 × 0.19 × 0.21 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 1.89 cm$^{-1}$ |
| Diffractometer, scan mode: | AFC10/Saturn724+, $\varphi/\omega$ |
| $2\theta_{max}$: | 54.92° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 9790, 2884 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 2184 |
| $N(param)_{refined}$: | 174 |
| Programs: | SHELXS-97, SHELXL-97 [7] |

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

| Atom | Site | *x* | *y* | *z* | $U_{iso}$ |
|---|---|---|---|---|---|
| H(2) | 8*f* | 0.8365 | 0.6419 | 0.6267 | 0.031 |
| H(3) | 8*f* | 0.9881 | 0.6141 | 0.5538 | 0.036 |
| H(4) | 8*f* | 0.9380 | 0.5898 | 0.4219 | 0.038 |
| H(5) | 8*f* | 0.7348 | 0.5882 | 0.3568 | 0.032 |
| H(8A) | 8*f* | 0.3522 | 0.6120 | 0.5564 | 0.029 |
| H(8B) | 8*f* | 0.3153 | 0.5929 | 0.4658 | 0.029 |
| H(9A) | 8*f* | 0.3670 | 0.7931 | 0.5320 | 0.026 |
| H(9B) | 8*f* | 0.3211 | 0.7714 | 0.4423 | 0.026 |
| H(3A) | 8*f* | –0.3788 | 0.4505 | 0.7180 | 0.042 |
| H(3B) | 8*f* | –0.2914 | 0.3928 | 0.6924 | 0.042 |
| H(5A) | 8*f* | –0.0897 | 0.4342 | 0.6898 | 0.049 |
| H(5B) | 8*f* | –0.1057 | 0.3298 | 0.6806 | 0.049 |
| H(6A) | 8*f* | 0.2351 | 0.6545 | 0.7135 | 0.065 |
| H(6B) | 8*f* | 0.1515 | 0.7327 | 0.7140 | 0.065 |
| H(1N) | 8*f* | 0.572(3) | 0.657(3) | 0.608(2) | 0.05(1) |
| H(2N) | 8*f* | 0.494(3) | 0.607(3) | 0.394(2) | 0.031(9) |
| H(4A) | 8*f* | 0.050(2) | 0.578(1) | 0.732(2) | 0.04(1) |

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

| Atom | Site | Occ. | *x* | *y* | *z* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| N(1) | 8*f* | | 0.5869(2) | 0.6396(2) | 0.5599(1) | 0.021(1) | 0.020(1) | 0.022(1) | –0.0001(9) | 0.0053(9) | 0.0007(9) |
| N(2) | 8*f* | | 0.5388(2) | 0.6146(2) | 0.4366(1) | 0.022(1) | 0.020(1) | 0.022(1) | –0.0023(9) | 0.001(1) | –0.0029(9) |
| C(1) | 8*f* | | 0.6962(2) | 0.6298(2) | 0.5340(2) | 0.020(1) | 0.015(1) | 0.025(1) | 0.002(1) | 0.006(1) | 0.003(1) |
| C(2) | 8*f* | | 0.8162(3) | 0.6308(2) | 0.5729(2) | 0.024(1) | 0.022(1) | 0.027(1) | 0.000(1) | –0.003(1) | 0.006(1) |
| C(3) | 8*f* | | 0.9048(3) | 0.6148(2) | 0.5293(2) | 0.019(1) | 0.021(1) | 0.050(2) | 0.003(1) | 0.004(1) | 0.007(1) |
| C(4) | 8*f* | | 0.8744(3) | 0.5997(2) | 0.4499(2) | 0.026(2) | 0.024(1) | 0.049(2) | 0.003(1) | 0.018(1) | –0.001(1) |
| C(5) | 8*f* | | 0.7549(3) | 0.5986(2) | 0.4107(2) | 0.032(2) | 0.023(1) | 0.029(2) | –0.002(1) | 0.012(1) | –0.007(1) |
| C(6) | 8*f* | | 0.6656(2) | 0.6138(2) | 0.4550(2) | 0.020(1) | 0.016(1) | 0.025(1) | –0.000(1) | 0.003(1) | –0.002(1) |
| C(7) | 8*f* | | 0.4951(2) | 0.6301(2) | 0.5006(2) | 0.021(1) | 0.014(1) | 0.028(1) | –0.001(1) | 0.004(1) | 0.001(1) |
| C(8) | 8*f* | | 0.3635(2) | 0.6374(2) | 0.5056(2) | 0.020(1) | 0.020(1) | 0.034(2) | –0.000(1) | 0.006(1) | 0.000(1) |
| C(9) | 8*f* | | 0.3159(2) | 0.7474(2) | 0.4945(2) | 0.018(1) | 0.019(1) | 0.029(1) | –0.003(1) | 0.006(1) | 0.001(1) |
| S(1) | 4*e* | | ½ | 0.63841(8) | ¾ | 0.0231(5) | 0.0318(6) | 0.0220(5) | 0 | 0.0070(4) | 0 |
| O(1) | 8*f* | 0.88(1) | 0.5141(5) | 0.7080(2) | 0.6861(2) | 0.054(3) | 0.035(1) | 0.033(2) | –0.005(1) | 0.017(2) | –0.002(1) |
| O(2) | 8*f* | 0.88 | 0.3910(3) | 0.5757(2) | 0.7259(3) | 0.033(2) | 0.041(2) | 0.068(3) | –0.011(1) | 0.001(2) | 0.010(2) |
| O(1') | 8*f* | 0.12 | 0.567(2) | 0.7051(4) | 0.705(1) | 0.054(3) | 0.035(1) | 0.033(2) | –0.005(1) | 0.017(2) | –0.002(1) |
| O(2') | 8*f* | 0.12 | 0.421(2) | 0.5702(4) | 0.695(1) | 0.033(2) | 0.041(2) | 0.068(3) | –0.011(1) | 0.001(2) | 0.010(2) |
| O(3) | 8*f* | | –0.3656(2) | 0.3958(2) | 0.6957(1) | 0.030(1) | 0.035(1) | 0.037(1) | 0.0022(9) | –0.0035(9) | –0.009(1) |
| O(4) | 4*e* | | 0 | 0.5414(2) | ¾ | 0.025(2) | 0.027(2) | 0.041(2) | 0 | 0.002(1) | 0 |
| O(5) | 8*f* | | –0.1353(2) | 0.3879(2) | 0.6664(1) | 0.036(1) | 0.030(1) | 0.051(1) | 0.002(1) | –0.005(1) | –0.008(1) |
| O(6) | 8*f* | | 0.1613(2) | 0.6708(2) | 0.7034(2) | 0.030(1) | 0.043(2) | 0.089(2) | 0.001(1) | 0.013(1) | 0.017(1) |

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