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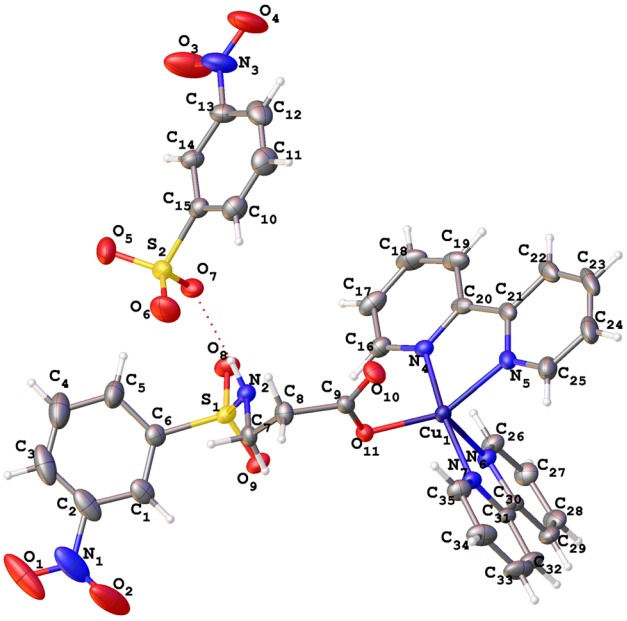
# Crystal structure of (((3-nitrophenyl)sulfonyl)-*β*-alaninato-*κO*)bis(2,2′-bipyridine-$\kappa^2N$, *N*′) copper(II) 3-nitrobenzenesulfonate, $C_{35}H_{29}CuN_7O_{11}S_2$



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**Abstract**

$C_{35}H_{29}CuN_7O_{11}S_2$, triclinic, $P\bar{1}$ (no. 2), $a$ = 12.2643(9) Å, $b$ = 13.3486(9) Å, $c$ = 13.5247(11) Å, $\alpha$ = 69.595(7)°, $\beta$ = 71.363(7)°, $\gamma$ = 67.275(7)°, $V$ = 1869.8(3) Å$^3$, $Z$ = 2, $R_{gt}(F)$ = 0.0570, w$R_{ref}(F^2)$ = 0.1781, $T$ = 293(2) K.

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A part of the molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

## 1 Source of material

*β*-Alanine (8.91 g, 100 mmol) and sodium hydroxide (8.00 g, 200 mmol) were dissolved in 100 mL of deionized water. Subsequently, *m*-nitrobenzenesulfonyl chloride (22.16 g, 100 mmol) was introduced into the reaction mixture at ambient temperature under constant stirring. After a reaction period of 10 h, the pH of the solution was carefully adjusted to 1 using a 6 mol/L hydrochloric acid solution. The resultant precipitate was then separated by filtration and subsequently dried to obtain a white solid product. Next, mix 316.9 mg of the aforementioned white solid with $Cu(CH_3COO)_2{\cdot}H_2O$ (99.82 mg, 0.5 mmol) and 2,2′-bipyridine (78.09 mg, 0.5 mmol), and dissolve them in 18 mL of a 1:1 methanol-water solution. Then, adjust the pH of the solution to approximately 7 using a 2 mol/L sodium hydroxide solution. Load the solution into a Teflon-lined reaction vessel and react at 80 °C for 12 h and then filter. Blue block crystals of the title compound were obtained by slow evaporation at room temperature within one week.

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

| | |
|---|---|
| Crystal: | Blue block |
| Size: | 0.68 × 0.36 × 0.34 mm |
| Wavelength: | MoKα radiation (0.71073 Å) |
| $\mu$: | 0.77 mm$^{-1}$ |
| Diffractometer, scan mode: | Multiwire proportional, $\varphi$ and $\omega$ |
| $\theta_{max}$, completeness: | 29.4°, >99 % |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 17121, 8714, 0.028 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\sigma(I_{obs})$, 5,797 |
| $N(param)_{refined}$: | 508 |
| Programs: | Bruker,[1] Olex2,[2] SHELX[3,4] |

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$).

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cu1 | 0.75955 (3) | 0.73506 (3) | 0.54720 (3) | 0.04072 (15) |
| S1 | 0.48513 (8) | 0.44550 (8) | 0.72757 (7) | 0.0466 (2) |
| O1 | 0.5261 (8) | 0.0130 (4) | 1.1074 (4) | 0.185 (3) |
| O2 | 0.6698 (7) | 0.0551 (5) | 0.9786 (7) | 0.180 (4) |
| O8 | 0.3993 (2) | 0.4928 (3) | 0.6607 (2) | 0.0670 (8) |
| O9 | 0.6089 (2) | 0.3940 (2) | 0.6827 (2) | 0.0598 (7) |
| O10 | 0.6117 (3) | 0.7882 (2) | 0.7380 (2) | 0.0642 (7) |
| O11 | 0.6981 (2) | 0.63453 (19) | 0.67902 (18) | 0.0444 (6) |
| N1 | 0.5620 (9) | 0.0747 (5) | 1.0257 (6) | 0.127 (3) |
| N2 | 0.4797 (3) | 0.5433 (3) | 0.7741 (2) | 0.0457 (7) |
| N4 | 0.6166 (2) | 0.7801 (2) | 0.4854 (2) | 0.0424 (6) |
| N5 | 0.7470 (3) | 0.9019 (2) | 0.4710 (2) | 0.0486 (7) |
| N6 | 0.8848 (2) | 0.6654 (2) | 0.4168 (2) | 0.0441 (7) |
| N7 | 0.9163 (3) | 0.6938 (2) | 0.5877 (2) | 0.0448 (7) |
| C1 | 0.5190 (5) | 0.2492 (4) | 0.8855 (4) | 0.0670 (12) |
| H1 | 0.601483 | 0.234490 | 0.855770 | 0.080* |
| C2 | 0.4762 (7) | 0.1772 (4) | 0.9760 (5) | 0.0915 (19) |
| C3 | 0.3539 (9) | 0.1967 (6) | 1.0224 (5) | 0.115 (3) |
| H3 | 0.327059 | 0.145885 | 1.083971 | 0.138* |
| C4 | 0.2753 (7) | 0.2909 (7) | 0.9762 (6) | 0.117 (2) |
| H4 | 0.192975 | 0.305074 | 1.006645 | 0.140* |
| H2 | 0.404 (6) | 0.582 (6) | 0.792 (5) | 0.140* |
| C5 | 0.3130 (4) | 0.3665 (4) | 0.8854 (4) | 0.0785 (14) |
| H5 | 0.257154 | 0.431663 | 0.854900 | 0.094* |
| C6 | 0.4356 (4) | 0.3446 (3) | 0.8397 (3) | 0.0543 (9) |
| C7 | 0.5619 (3) | 0.5143 (3) | 0.8468 (3) | 0.0473 (8) |
| H7A | 0.641606 | 0.467946 | 0.818778 | 0.057* |
| H7B | 0.531100 | 0.472014 | 0.917996 | 0.057* |
| C8 | 0.5703 (4) | 0.6204 (3) | 0.8544 (3) | 0.0529 (9) |
| H8A | 0.616000 | 0.601223 | 0.908764 | 0.063* |
| H8B | 0.489340 | 0.667259 | 0.878847 | 0.063* |
| C9 | 0.6291 (3) | 0.6885 (3) | 0.7498 (3) | 0.0420 (8) |
| C16 | 0.5565 (3) | 0.7105 (4) | 0.4972 (3) | 0.0537 (9) |
| H16 | 0.575440 | 0.639097 | 0.543988 | 0.064* |
| C17 | 0.4668 (4) | 0.7427 (5) | 0.4413 (4) | 0.0722 (14) |
| H17 | 0.426757 | 0.693076 | 0.448340 | 0.087* |
| C18 | 0.4379 (4) | 0.8503 (5) | 0.3746 (4) | 0.0822 (16) |
| H18 | 0.376695 | 0.874204 | 0.337305 | 0.099* |
| C19 | 0.4984 (4) | 0.9211 (5) | 0.3634 (3) | 0.0709 (13) |
| H19 | 0.479265 | 0.993342 | 0.318145 | 0.085* |
| C20 | 0.5884 (3) | 0.8854 (3) | 0.4195 (3) | 0.0506 (9) |
| C21 | 0.6595 (3) | 0.9542 (3) | 0.4145 (3) | 0.0508 (9) |
| C22 | 0.6398 (5) | 1.0663 (4) | 0.3555 (3) | 0.0730 (14) |
| H22 | 0.579670 | 1.102144 | 0.315675 | 0.088* |
| C23 | 0.7127 (6) | 1.1225 (4) | 0.3582 (4) | 0.0916 (19) |
| H23 | 0.701026 | 1.197542 | 0.320260 | 0.110* |
| C24 | 0.8001 (5) | 1.0696 (4) | 0.4151 (5) | 0.0868 (17) |
| H24 | 0.849610 | 1.106955 | 0.416380 | 0.104* |
| C25 | 0.8149 (4) | 0.9585 (4) | 0.4717 (4) | 0.0677 (12) |
| H25 | 0.874785 | 0.921995 | 0.511871 | 0.081* |
| C26 | 0.8626 (4) | 0.6534 (4) | 0.3324 (3) | 0.0576 (10) |
| H26 | 0.782372 | 0.671308 | 0.329256 | 0.069* |
| C27 | 0.9522 (4) | 0.6159 (4) | 0.2490 (3) | 0.0648 (11) |
| H27 | 0.933401 | 0.607934 | 0.191256 | 0.078* |
| C28 | 1.0703 (4) | 0.5907 (4) | 0.2547 (4) | 0.0681 (12) |
| H28 | 1.133175 | 0.565856 | 0.199700 | 0.082* |
| C29 | 1.0953 (3) | 0.6020 (3) | 0.3406 (3) | 0.0582 (10) |
| H29 | 1.175059 | 0.583848 | 0.345148 | 0.070* |
| C30 | 1.0010 (3) | 0.6407 (3) | 0.4211 (3) | 0.0430 (8) |
| C31 | 1.0183 (3) | 0.6564 (3) | 0.5168 (3) | 0.0455 (8) |
| C32 | 1.1310 (3) | 0.6335 (4) | 0.5367 (4) | 0.0663 (12) |
| H32 | 1.201320 | 0.609983 | 0.486838 | 0.080* |
| C33 | 1.1369 (4) | 0.6462 (4) | 0.6315 (5) | 0.0794 (15) |
| H33 | 1.211584 | 0.631119 | 0.646165 | 0.095* |
| C34 | 1.0331 (4) | 0.6807 (4) | 0.7038 (5) | 0.0769 (14) |
| H34 | 1.036391 | 0.687901 | 0.768673 | 0.092* |
| C35 | 0.9236 (4) | 0.7049 (4) | 0.6800 (4) | 0.0598 (10) |
| H35 | 0.852748 | 0.729566 | 0.728982 | 0.072* |
| S2 | 0.16580 (9) | 0.69617 (9) | 0.93808 (8) | 0.0590 (3) |
| O3 | −0.1695 (6) | 0.9855 (5) | 0.7352 (6) | 0.170 (3) |
| O4 | −0.1665 (5) | 1.1528 (4) | 0.7106 (6) | 0.172 (3) |
| O5 | 0.0617 (3) | 0.6572 (3) | 0.9878 (3) | 0.0928 (11) |
| O6 | 0.2433 (4) | 0.6721 (3) | 1.0076 (3) | 0.1040 (13) |
| O7 | 0.2293 (2) | 0.6640 (2) | 0.8381 (2) | 0.0656 (8) |
| N3 | −0.1312 (5) | 1.0508 (5) | 0.7459 (5) | 0.1149 (19) |
| C10 | 0.1539 (4) | 0.9160 (5) | 0.9087 (4) | 0.0722 (13) |
| H10 | 0.218702 | 0.885615 | 0.942951 | 0.087* |
| C11 | 0.1092 (5) | 1.0302 (5) | 0.8710 (5) | 0.0888 (16) |
| H11 | 0.143064 | 1.076719 | 0.880609 | 0.107* |
| C12 | 0.0154 (5) | 1.0759 (4) | 0.8194 (5) | 0.0849 (15) |
| H12 | −0.014609 | 1.153344 | 0.792551 | 0.102* |
| C13 | −0.0340 (4) | 1.0049 (4) | 0.8078 (4) | 0.0684 (12) |
| C14 | 0.0080 (3) | 0.8911 (3) | 0.8462 (3) | 0.0572 (10) |
| H14 | −0.027630 | 0.844877 | 0.838393 | 0.069* |
| C15 | 0.1041 (3) | 0.8458 (3) | 0.8966 (3) | 0.0513 (9) |

## 2 Experimental details

Hydrogen atoms were added using riding models. Their $U_{iso}$ values were set to 1.2$U_{eq}$ of the parent atoms. The structure was solved with the ShelXT[3] structure solution program and refined with the ShelXL.[4]

## 3 Comment

Sulfonated amino acids are a class of amino acid derivatives formed by the substitution of a sulfonate group on the nitrogen atom of the amino group. These compounds have shown a broad potential for application in the fields of biochemistry and materials science due to their unique chemical properties.[5–8] Sulfonated amino acids can participate in coordination not only through the oxygen atom and the nitrogen atom of their amino acid group but also the oxygen atoms of their sulfonate group can form

coordination bonds with metal ions.[9–11] This dual coordination capability makes sulfonated amino acids ideal candidates for constructing new metal complexes. These complexes may exhibit a variety of coordination geometries. In our group, a series of sulfonylated amino acid complexes have been successfully synthesized.[12–15] This contribution is part of our ongoing research focus on the complexes of sulfonylated amino acids.

In this paper, the synthesis and crystal structure of a new copper(II) complex with ((3-nitrophenyl)sulfonyl)-*β*-alanine and 2,2′-bipyridine were reported. Single crystal X-ray structure analysis reveals that the title complex crystallizes in the triclinic system with space group $P\bar{1}$. The asymmetric unit is composed of an independent Cu(II) ion, one ((3-nitrophenyl)sulfonyl)-*β*-alanine ligand, two 2,2′-bipyridine ligands, and one 3-nitrobenzenesulfonate. The crystallographically unique Cu(II) ion of the title complex is five-coordinated by four N atoms (N4, N5, N6, N7) from two 2,2′-bipyridine groups, one O atom (O11) from ((3-nitrophenyl)sulfonyl)-*β*-alanine group, giving rise to a hexahedral geometry. Cu–N bond lengths fall in the range 1.977(3)–2.155(3) Å, and Cu–O bond length is 1.950(2) Å. The bond lengths observed fall within the expected range and align closely with those documented for analogous complexes in prior studies.[12,13,16] The extended three-dimensional supramolecular network is constructed through hydrogen bond N2–H2⋯O7, and *π*–*π* interactions between the aromatic rings of 3-nitrobenzenesulfonate and 2,2′-bipyridine.

**Author contributions:** All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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**Conflict of interest statement:** The authors declare no conflicts of interest regarding this article.

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