

Panče Naumov,^{a*} Gligor Jovanovski^a and Seik Weng Ng^b^aInstitute of Chemistry, Faculty of Science, 'Sv. Kiril i Metodij' University, PO Box 162, MK-1001 Skopje, Macedonia, and ^bInstitute of Postgraduate Studies, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: pance@chem.titech.ac.jp

Key indicators

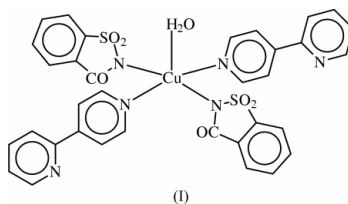
Single-crystal X-ray study
T = 95 K
Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$
R factor = 0.048
wR factor = 0.104
Data-to-parameter ratio = 17.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Aquabis(2,4'-bipyridine)di(o-sulfobenzimidato)copper(II) at 95 K

The square-pyramidal environment of the metal in the copper(II) saccharinate neutral complex with 2,4'-bipyridine, $[\text{Cu}(\text{C}_7\text{H}_4\text{NO}_3\text{S})_2(\text{C}_{10}\text{H}_8\text{N}_2)_2(\text{H}_2\text{O})]$, consists of *trans*-oriented *N*-saccharinate and 2,4'-bipyridine ligand pairs and an apical aqua molecule. A crystallographic twofold rotation axis passes through Cu and the aqua ligand.

Comment

The deprotonated anion of saccharin (*o*-sulfobenzimide) binds to a plethora of metal cations to form compounds whose structures can be conveniently probed by infrared spectroscopy (Naumov & Jovanovski, 2000, 2001). The compounds are coordinatively unsaturated; among these, the copper(II) derivative has been well documented to form adducts, particularly with *N*-heterocycles (Naumov, Jovanovski, Drew *et al.*, 2001; Naumov, Jovanovski, Ristova *et al.*, 2001). On the other hand, one *N*-heterocycle that has not been used for binding is the 4,4'-bipyridine ligand; in our study, the spacer ligand afforded a polymeric structure displaying a rhombic grid that encapsulates a dichloromethane molecule (Naumov, Jovanovski, Hanna *et al.*, 2001). We then extended this study to the 2,4'-bipyridine analog, which should adopt a helical conformation if a similar metal–ligand bonding is retained, as we noted in other systems; the 4,4'-isomer yields layer motifs whereas the 2,4'-isomer is able to furnish chains (Fun *et al.*, 1999).



Coordination through the 2-*N* atom is not favored in the title compound, (I), and the ligand coordinates instead only through the 4-*N* atom (Fig. 1). The fifth coordination site of the square-pyramidal geometry with *trans*-oriented pairs of *N*-saccharinate and bipyridine ligands is taken up by a water molecule.

Experimental

Tetraaquabis(*o*-sulfobenzimidato)copper(II) dihydrate was synthesized from the reaction of copper(II) nitrate and sodium *o*-sulfobenzimidate in water in high yield (Naumov & Jovanovski, 2001). A small quantity of the compound (*ca* 0.05 g) was dissolved in warm ethanol (50 ml). An amount of 2,4'-bipyridine corresponding to twice the amount of the copper compound was dissolved in dichloro-

Received 14 November 2001

Accepted 3 December 2001

Online 14 December 2001

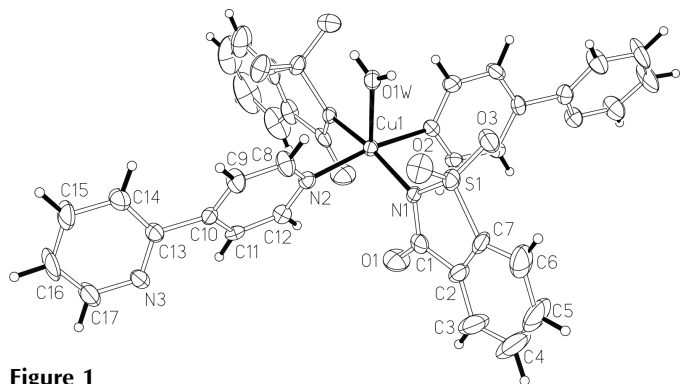


Figure 1
ORTEP II (Johnson, 1976) plot with ellipsoids at the 50% probability level.

methane (5 ml) and the solution was layered over 10 ml portions of a filtered solution of the copper reagent. Individual attempts invariably resulted in a small amount of wing-shaped crystals that were mixed with regularly shaped crystals. Both types gave the same elemental analyses. The diffraction measurements were carried out on a regularly shaped specimen. Elemental CHN analysis (calculated/found): C 53.85/53.77, H 3.46/3.27, N 11.08/10.86%.

Crystal data

[Cu(C₇H₄NO₃S)₂(C₁₀H₈N₂)₂(H₂O)]
M_r = 758.27
 Monoclinic, C₂
a = 12.5758 (3) Å
b = 12.2526 (2) Å
c = 11.6563 (3) Å
 β = 101.140 (1)°
V = 1762.23 (7) Å³
Z = 2

D_x = 1.429 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 5360 reflections
 θ = 1.8–27.6°
 μ = 0.79 mm⁻¹
T = 95 (2) K
 Plate, blue
 0.18 × 0.18 × 0.04 mm

Data collection

Siemens CCD area-detector diffractometer
 ω scans
 Absorption correction: empirical (SADABS; Sheldrick, 1996)
T_{min} = 0.870, *T_{max}* = 0.969
 8924 measured reflections

4029 independent reflections
 3056 reflections with *I* > 2σ(*I*)
R_{int} = 0.094
 θ_{\max} = 27.6°
h = −16 → 16
k = −15 → 15
l = −14 → 15

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.048
wR(*F*²) = 0.104
S = 0.95
 4029 reflections
 231 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0197P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.56 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.94 \text{ e } \text{Å}^{-3}$
 Absolute structure: Flack (1983)
 Flack parameter = 0.34 (2)

Table 1

Selected geometric parameters (Å, °).

Cu1—N1	1.999 (3)	Cu1—O1w	2.272 (4)
Cu1—N2	2.027 (3)		
N1—Cu1—N1 ⁱ	168.2 (2)	N1—Cu1—O1w	95.9 (1)
N1—Cu1—N2	89.8 (1)	N2—Cu1—N2 ⁱ	168.6 (2)
N1—Cu1—N2 ⁱ	89.0 (1)	N2—Cu1—O1w	95.7 (1)

Symmetry code: (i) 1 − *x*, *y*, 2 − *z*.

Of the 4029 reflections, 2139 were Friedel pairs. The water H atom was located and refined. The other H atoms were positioned geometrically, with *U*(H) = 1.2*U*_{eq}(C). The elemental CHN analysis confirmed the composition, eliminating the possibility of residual solvent molecules in the solvent-accessible voids. The somewhat larger ADP max/min ratio for C4 and C14 is an artifact of the quality of the crystallographic data.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP II (Johnson, 1976); software used to prepare material for publication: SHELXL97.

We thank Professor Y. Ohashi (Tokyo Institute of Technology) for kindly providing access to the CCD diffractometer. This work was supported by the National Science Council for R&D (IRPA 305/PFIZIK/610961) and the University of Malaya (F0758/2001A).

References

- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
 Fun, H.-K., Shanmuga Sundara Raj, S., Xiong, R.-G., Zuo, J.-L., Yu, Z., Zhu, X.-L., You, X.-Z. (1999). *J. Chem. Soc. Dalton Trans.* pp. 1711–1712.
 Johnson, C. K. (1976). ORTEP II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Naumov, P. & Jovanovski, G. (2000). *Struct. Chem.* **11**, 19–33.
 Naumov, P. & Jovanovski, G. (2001). *Curr. Org. Chem.* **5**, 1059–1077.
 Naumov, P., Jovanovski, G., Drew, M. G. B. & Ng, S. W. (2001). *Inorg. Chim. Acta*, **314**, 154–162.
 Naumov, P., Jovanovski, G., Ristova, M., Ibrahim, A. R., Cakir, S., Chantrapromma, S., Fun, H.-K. & Ng, S. W. (2001). Unpublished results.
 Naumov, P., Jovanovski, G., Hanna, V. J., Ibrahim, R. A., Chantrapromma, S., Fun, H.-K. & Ng, S. W. (2001). *Inorg. Chem. Commun.* In the press.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Siemens (1996). SMART and SAINT. Siemens AXS Inc., Madison, Wisconsin, USA.
 Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.