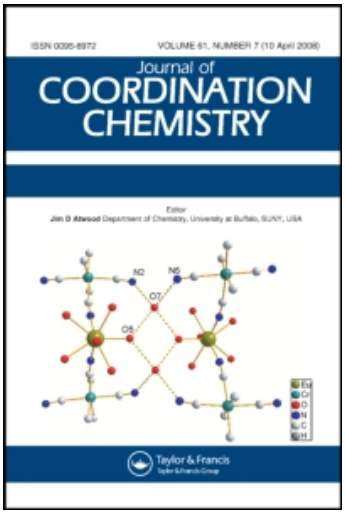

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Crystal structure of a new complex of [3-(1H-benzimidazol-yl)propionato-N][3-(1H-benzimidazol-yl) propionic acid-N]silver(I), $C_{20}H_{19}AgN_4O_4$

Ming-Hua Zeng[a]; Min-Xia Yao[a]; Hong Liang[a]; Seik Weng Ng[b]
[a] Department of Chemistry and Chemical Engineering, Guangxi Normal University, Guilin, Guangxi, People's Republic of China [b] Department of Chemistry, University of Malaya, Kuala Lumpur, Malaysia

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Crystal structure of a new complex of [3-(1*H*-benzimidazol-yl) propionato-*N*][3-(1*H*-benzimidazol-yl) propionic acid-*N*]silver(I), $C_{20}H_{19}AgN_4O_4$

MING-HUA ZENG†, MIN-XIA YAO†, HONG LIANG*† and SEIK WENG NG‡

†Department of Chemistry and Chemical Engineering, Guangxi Normal University, Guilin, Guangxi, 541004, People's Republic of China
‡Department of Chemistry, University of Malaya, Kuala Lumpur 50603, Malaysia

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The silver and acid hydrogen atoms in the crystal structure of $[Ag(pa)(Hpa)]_n$ (Hpa = 3-(1*H*-benzimidazol-2-yl) propionic acid-*N*) both lie on special positions of −1 site symmetry; the silver atom shows linear coordination [Ag–N = 2.109(3) Å, N–Ag–N = 180°]. The 'acid hydrogen' links molecules into a linear chain, and hydrogen bonds between the nitrogen-bound hydrogen atom and the carbonyl oxygen atom of an adjacent chain furnish a three-dimensional supramolecular structure. The compound, $C_{20}H_{19}AgN_4O_4$, belongs to the triclinic space group $P\bar{1}$ [$a = 6.536(7)$, $b = 8.127(9)$, $c = 9.051(1)$ Å; $\alpha = 81.692(2)$, $\beta = 82.819(2)$, $\gamma = 87.229(2)°$], and there is one formula unit in the unit cell.

Keywords: Silver; Linear coordination; Hydrogen bonds; A linear chain

1. Introduction

Noncovalent interactions play a central role in supramolecular self-assembly [1, 2]. Formation of a supramolecule occurs through hydrogen bonding, $\pi \cdots \pi$ stacking and other weak interactions between molecules [1]. Recently, more and more supramolecular compounds containing polymeric Ag(I) species have been described due to the coordination modes of silver(I). For instance, Ag can form linear, trigonal, tetrahedral and hexagonal coordination compounds. Here, we report a new supramolecular compound from self-assembly of silver(I) with 3-(1*H*-benzimidazol-2-yl) propionic acid-*N*.

*Corresponding author. Tel.: +86-773-3308-568. Fax: +86-773-5846-297. Email: zmh@mailbox.gxnu.edu.cn

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2. Experiment

2.1. *Materials and instrumentation*

All solution and chemicals were commercial reagents and used without further purification. Elemental analyses (C, H, N) were performed on a Perkin–Elmer 2400II elemental analyzer. Infrared spectra were recorded in KBr discs from 4000–400 cm^{-1} on a Perkin–Elmer Spectrum One FT/IR spectrometer. The crystal structure was determined by a Bruker APEX area-detector diffractometer with SHELXLTL software.

2.2. *Synthesis of [Ag(pa)(Hpa)]*

3-(1*H*-benzimidazol-2-yl) propionic acid (Hpa) was prepared according to an improved literature procedure [3]. *o*-Phenylenediamine (5.00 g, 46 mmol) and succinic acid (5.46 g, 46 mmol) were dissolved in 10 mL of polyphosphoric acid (PPA) and 40 mL of phosphoric acid; the mixture was heated for 4 h at 458 K with stirring. Then the reaction mixture was poured into 200 mL of water after cooling to 373 K. The precipitate (A) was filtered, and the filtrate gave a white solid (B) after cooling overnight. The solid B was filtered, recrystalized from hot water, and shown to be the Hpa product, 3.18 g (36% yield). FTIR (KBr): 3428, 3160, 1673, 1632, 1575, 1523, 1490, 1461, 1293 cm^{-1}.

A stirred mixture of silver nitrate (0.042 g, 0.25 mmol) and Hpa (0.095 g, 0.5 mmol) in water (10 mL) was treated with triethylamine (0.2 mL), then sealed in a 15-mL Teflon-lined stainless steel autoclave and heated at 393 K for 72 h, giving rise to colorless crystals of **1** with 39% yield. Elemental analysis: Anal. Calcd for $C_{20}H_{19}AgN_4O_4$: C 49.30, H 3.93, N 11.50%; found: C 49.19, H 3.41, N 3.84%.

2.3. *X-ray crystallography*

Cystallographic data for **1** were collected with a Bruker APEX area detector diffractometer equipped with graphite-monochromatized Mo-Kα X-ray radiation ($\lambda = 0.71073$ Å) using the ω-θ scan mode in the range $2.3 \leq \theta \leq 26.0^\circ$ at 293 K. Raw frame data were integrated with the SAINT [4] program. The structure was solved by direct methods using SHELXS-97 and refined by full-matrix least-squares on F^2 using SHELXS-97 [5]. An absorption correction was applied with the program SADABS [6]. All nonhydrogen atoms were refined anisotropically. Hydrogen atoms were set in calculated positions and refined by a riding mode, with a common thermal parameter. All calculations and graphics were performed with SHELXTL [5].

All non-H atoms were refined anisotropically and the hydrogen atoms were located on the calculated positions and refined isotropically to final R indices (all data) of R_1 (0.042) and wR_2 (0.076).

Crystallographic data for complex **1**: Crystal dimensions $0.20 \times 0.16 \times 0.08$ mm^3. $C_{20}H_{19}AgN_4O_4$, Mr = 487.26, Triclinic, $P\bar{1}$, $a = 6.536(7)$, $b = 8.127(9)$, $c = 9.051(1)$ Å, $\alpha = 81.692(2)$, $\beta = 82.819(2)$, $\gamma = 87.229(2)^\circ$, $V = 471.72(9)$ $Å^3$, $Z = 1$, $D_c = 1.715$ g cm^{-3}, $\mu = 1.105$ mm^{-1}, $R_1 = 0.034$, $wR_2 = 0.076$ [$I > 2\sigma(I)$], GooF = 1.03. Some 2631 reflections were collected, with 1812 independent reflections ($R_{int} = 0.016$). The largest peak

and hole on the final difference-Fourier map are 0.57 and $-0.30\,e\,Å^{-3}$. CCDC deposition number: 294698.

3. Results and discussion

The title compound exists as a hydrogen-bonded $[Ag(pa)(Hpa)]_n$ chain; the structure represents one of the few structurally verified metal derivatives of (1*H*-benzimidazolyl)-3-propionic acid. A copper complex is also known; in the compound, the carboxylate binds to copper through the carboxyl [7]. The present silver derivative possesses an inversion center at Ag atom (figure 1), and Ag is coordinated by two nitrogen atoms from pa and Hpa in a linear geometry with the Ag–N distance of 2.109(3) Å. The Ag–N distance is comparable with distances found in $[Ag_2(psb)_2](ClO_4)_2$ (psb = 1,2-*bis*[(4-pyridinyl)-sulfanylmethyl] benzene) (2.120 ~ 2.148 Å) [8]. The complex features an 'acid hydrogen' that lies midway between two adjacent single-bond carboxylate oxygen atoms [2.476(4) Å], giving rise to formation of a linear chain (figure 2). Adjacent hydrogen-bonded chains are linked into the three-dimensional network by N–H ··· O strong hydrogen bonding with the distance of 2.744(4) Å (figure 3). It should be noted that the structure is further stabilized by the $\pi \cdots \pi$ stacking interactions of benzimidazole rings with face-to-face distance of 3.44 Å, comparable

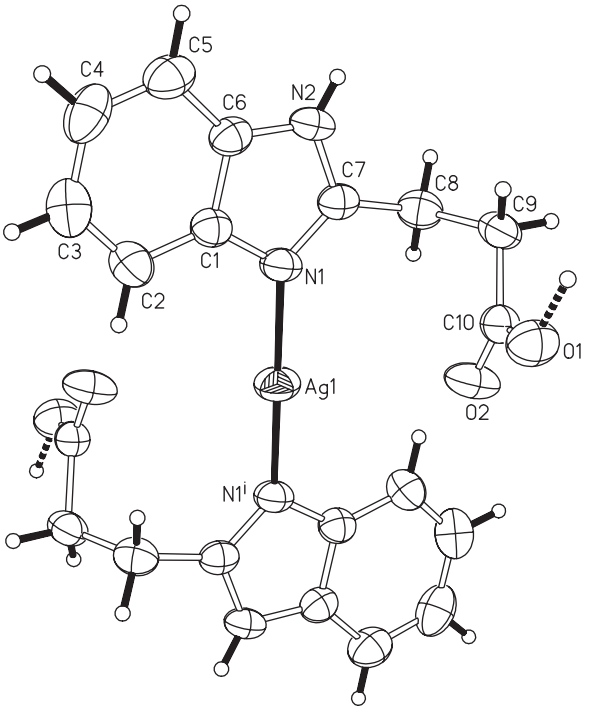

Figure 1. ORTEP view of the discrete unit of $[Ag(pa)(Hpa)]_n$ along with atom numbering scheme.

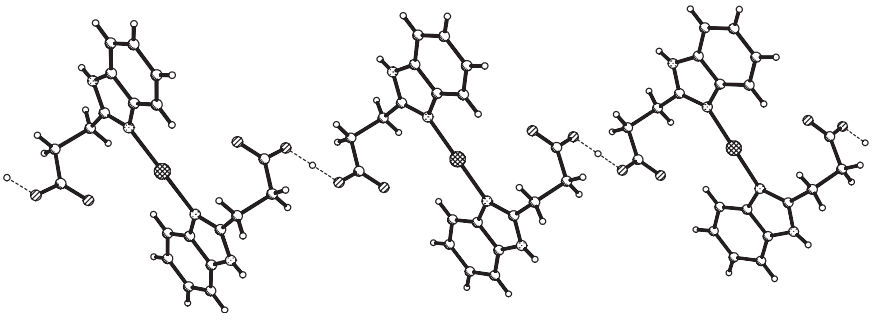

Figure 2. The hydrogen-bonded chain.

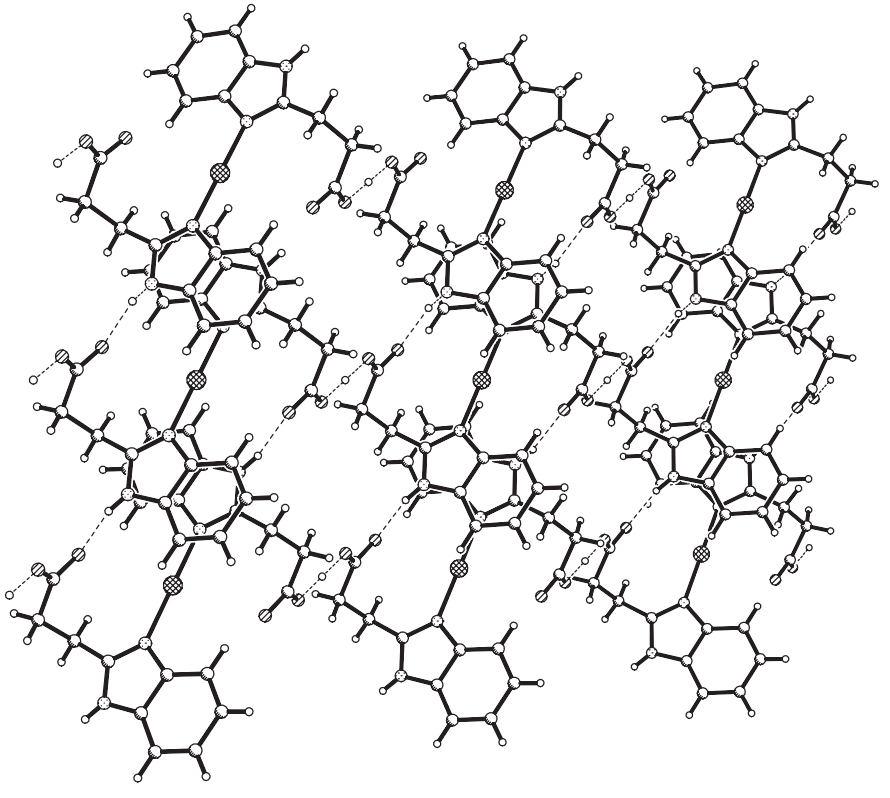

Figure 3. Stacking diagram of the title compound **1**.

with that of [Ag(μ_3-hmt)](ns) · 0.5(benzene) · $2H_2O$(hmt = hexamethylenetramine, ns = 2-naphthalenesulfonate) [9].

Acknowledgements

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References

[1] G.R. Desiraju. *Angew. Chem. Int. Ed. Engl.*, **34** (1995).
[2] C.B. Aakeröy, K.R. Seddon. *Chem. Soc. Rev.*, **6**, 397 (1993).
[3] M.A. Phillips. *J. Chem. Soc.*, 2393 (1928).
[4] Siemens. *SAINT: Siemens Area DetectorIntegration Software*, Siemens Analytical X-ray instruments Inc., Madison, WI, USA (1996).
[5] G.M. Sheldrick. *SHELXTL Software Reference Manual, Version 5.1, Brucker AXS Inc*, Madison, WI, USA (1997).
[6] G.M. Sheldrick, *SADABS. Program for Empirical Absorption Correction of Area Detector Data*, University of Göttingen, Germany (1996).
[7] K.Y. Choi. *J. Chem. Cryst.*, **34**, 603 (2004).
[8] Y.J. Zhao, M.C. Hong, W.P. Su, R. Cao, Z.Y. Zhou, A.S.C. Chan. *Chem. Lett.*, **29**(1), 28 (2000).
[9] S.L. Zheng, M.L. Tong, X.M. Chen, S.W. Ng. *J. Chem. Soc., Dalton Trans.*, 360 (2002).