

银-烯配位的银(I)配合物的合成、晶体与荧光性质

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摘要: 以烟酸(4-烯丙基-2-甲氧基)苯酯(L)为配体, 在四氢呋喃/水体系中设计合成了银-双键配位的新型配合物 $[\text{AgL}(\text{NO}_3)]_n$ (**1**)。通过 X-射线单晶衍射方法对 L 和 **1** 进行了表征, 分析了其光谱及结构特征。结构分析表明, 化合物 L 和 **1** 都为单斜晶系, 配体 L 通过 C-H \cdots O 氢弱作用依次形成二聚体、二维层结构。配合物 **1** 中, 含有通过银-双键和氮原子配位的左旋曲绕链和通过银-氧弱作用形成的右旋曲绕链, 两种链通过 $\cdots\text{ABAB}\cdots$ 堆积形成二维结构, 最后通过 C-H \cdots O 弱作用形成三维结构。同时对化合物 L 和 **1** 的荧光性质进行了研究。

关键词: 烟酸(4-烯丙基-2-甲氧基)苯酯; 银-烯炔作用; 银配合物; 弱作用; 晶体结构

中图分类号: O614.122

文献标识码: A

文章编号: 1001-4861(2015)12-2291-07

DOI: 10.11862/CJIC.2015.304

Syntheses, Crystals and Luminescence of Silver(I) Complexes with Metal-Olefin Binding Interactions

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Abstract: One new alkene complex of silver(I), $[\text{AgL}(\text{NO}_3)]_n$ (**1**), (L=4-allyl-2-methoxyphenyl nicotinate) has been prepared by self-assembly of AgNO_3 with 4-allyl-2-methoxyphenyl nicotinate (L) in THF/ H_2O system. The structures of compounds L and **1** are determined by X-ray diffraction analysis. The crystals of the two compounds belong to the monoclinic space group. Compound L is connected to form a dimer, 2D layer sequentially by the weak C-H \cdots O hydrogen bonds. The structure of **1** consists of 1D left-handed helical chains of $[\text{AgL}]_\infty$ in which the allyl arms and N atoms are coordinated to silver atoms, and right-handed helical chains of $[\text{AgNO}_3]_\infty$ connecting through the Ag-O weak interactions. Two kinds of helices are connected in $\cdots\text{ABAB}\cdots$ fashion to form a 2D achiral layer. Finally, the resulted 2D net extends to the 3D supramolecular architecture by the weak C-H \cdots O interactions. In addition, the photoluminescence of L and **1** has been investigated in detail. CCDC: 985940, L; 985941, **1**.

Key words: 4-allyl-2-methoxyphenyl nicotinate; silver-alkene interaction; silver complex; weak interactions; crystal structure

0 Introduction

Metallosupramolecular chemistry involves the use of bridging organic ligands and metal ions for the construction of discrete and polymeric assemblies with

diverse molecular architectures^[1]. There has been rapid and popular development of Ag(I) complexes in recent years, as its structural versatility in its coordination chemistry^[2]. First, Ag(I) is able to form bonds with different donors simultaneously, which readily binds

收稿日期: 2015-06-18。收修改稿日期: 2015-09-29。

浙江省教育厅项目(No.Y201430862, FX2013215), 2014 年浙江省大学生科技创新活动计划暨新苗人才计划项目(No.2014R465011)资助。

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to nitrogen heterocycles, as well as to ligands having oxygen, phosphorus, sulfur and other common donors^[3-8], and Ag(I) has long been known to interact with carbon-carbon double bonds^[1], too. Second, the coordination geometries of Ag(I) are a wide variety and offer particularly versatile structures. Third, the d^{10} - d^{10} interactions between two close-shells of Ag(I) atom may exist in the complexes of Ag(I), increasing the possibility of forming complicated multi-dimensional systems^[9]. So Ag(I) is a favorable building block or connecting node for coordination polymers, which have special photophysical and photochemical properties^[10].

Furthermore, luminescent compounds composed of d^{10} metal centers and organic ligands are of great interest because of their potential applications in the areas of chemical sensors and photoluminescent properties^[11], such as light-emitting diodes (LEDs)^[12], sensors, photochemistry^[13].

Herein, 4-allyl-2-methoxyphenyl nicotinate was chosen to be the ligand to generate metallacyclic ensembles because of its structures of the pyridine ring, which has the better coordination ability, and the carbon-carbon double bonds, which can interact with Ag(I) to form the interesting metal organic complexes.

1 Experimental

1.1 Materials

All reagents and solvents employed were commercially available and used without further purification, except acetone and dichloromethane were used after dried by MgSO_4 .

1.2 Physical measurement

Powder X-ray diffraction measurements were carried out with a Bruker D8 Focus X-ray diffractometer to check the phase purity. The C, H and N microanalyses were performed with Perkin Elmer 2400 II CHNO/S elemental analyzer. The FT-IR spectra were recorded from KBr pellets in the range of $4\,000\sim 400\text{ cm}^{-1}$ on a Shimadzu FTIR-8900 spectrometer. Thermogravimetric measurements were carried out from R.T. to $900\text{ }^\circ\text{C}$ on pre weighed samples in nitrogen stream using a Seiko Exstar 6000 TG/DTA 6300 apparatus with a heating rate of $10\text{ }^\circ\text{C}\cdot\text{min}^{-1}$.

The fluorescence on solid samples were determined with a RF-5301PC spectrophotometer.

1.3 Preparation of ligands and complex

1.3.1 Synthesis of 4-allyl-2-methoxyphenyl nicotinate (L)

The ligand L (4-allyl-2-methoxyphenyl nicotinate) was synthesized according to the literature^[14]. Thionyl chloride (150 mL) was added to 3-pyridine carboxylic acids (0.65 mol, 80 g). The mixture was stirred and refluxed for 3 h. The thionyl chloride was then evaporated under reduced pressure giving a less yellow crystalline residue of nicotinoyl hydrochloride. The purity of nicotinoyl hydrochloride was quite sufficient to use the product directly for the following synthesis.

A mixture solution of dry triethylamine (30 mL) and CH_2Cl_2 (100 mL) containing eugenol (16.4 g, 0.1 mol) was added dropwisely to a stirred solution of nicotinoyl hydrochloride (17.8 g, 0.1 mol) in dry dichloromethane (50 mL) at room temperature. After 3 h, to the mixture, saturated sodium carbonate was added dropwisely to adjust the pH value to about 7.0. The CH_2Cl_2 layer was washed successively with saturated NaCl solution and H_2O for three times. The CH_2Cl_2 layer was dried by anhydrous MgSO_4 and the solvent was removed under reduced pressure. Recrystallization in ethanol gave white prism crystals. Yield: 16.1 g (60%). m.p. $70\sim 72\text{ }^\circ\text{C}$. Anal. Calcd. for $\text{C}_{16}\text{H}_{15}\text{NO}_3$ (%): C, 71.36; H, 5.61; N, 5.20. Found(%): C, 71.35, H, 5.62, N, 5.21. IR (KBr, cm^{-1}): 3 130 (s, br), 1 745 (vs), 1 642(m), 1 606 (s), 1 589 (s), 1 508 (s), 1 464 (s), 995 (m), 911 (m), 734(s). ^1H NMR (400 MHz, CDCl_3): δ 3.39 (d, 2, $-\text{CH}_2-$), 3.80 (s, 3, $-\text{OCH}_3$), 5.09~5.14 (m, 2, $\text{H}_2\text{C}=\text{}$), 5.92~6.03(m, 1, $=\text{CH}$), 6.81 (d, 1, H-5), 6.84 (s, 1, H-3), 7.06 (d, 1, H-6), 7.42~7.45 (dd, 1, H-5'), 8.45 (d, 1, H-4'), 8.83 (d, H-6'), 9.39 (s, 1, H-2').

1.3.2 Synthesis of $[\text{AgL}(\text{NO}_3)]_n$ (1)

A solution of AgNO_3 (16.9 mg, 0.1 mmol) in 4.0mL water was slowly added to a solution of 4-allyl-2-methoxyphenyl nicotinate (L) (26.0 mg, 0.1 mmol) in THF (6 mL). Colorless single crystals suitable for X-ray analysis were obtained after a few days with

82% yield (based on L). Anal. Calcd. for $C_{16}H_{15}N_2O_6Ag$ (%): C, 43.76; H, 3.44; N, 6.38. Found (%): C, 43.80, H, 3.43, N, 6.40. IR (KBr, cm^{-1}): 3 448(br), 1 736 (s, br), 1 745 (s), 733(s).

1.4 X-ray crystallography

Crystallographic data were collected on a Rigaku R-Axis Rapid IP X-ray diffractometer with graphite-monochromated Mo $K\alpha$ radiation ($\lambda=0.071\ 073\ nm$). The data were corrected for Lp and absorption effects. The structures were solved by direct methods and expanded with difference Fourier techniques. All non-

hydrogen atoms were refined anisotropically in full-matrix least-squares refinements based on F^2 . All hydrogen atoms were generated geometrically. The C2 atom of allyl in L was disordered and the occupancies were rene'd to 1:1.07, respectively. All calculations were performed with SHELXTL-97 packages^[15]. The crystal data and structure refinements are summarized in Table 1, and the selected bond lengths and bond angles are listed in Table 2.

CCDC: 985940, L; 985941, **1**.

Table 1 Crystal data and structure and refinement for compounds L and **1**

Compounds	L	1
Empirical formula	$C_{16}H_{15}NO_3$	$C_{16}H_{15}AgN_2O_6$
Formula weight	269.29	439.17
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/c$
a / nm	1.117 9(2)	1.503 6(3)
b / nm	0.950 07(19)	0.698 6(1)
c / nm	1.395 3(3)	1.610 5(3)
$\beta / (^\circ)$	107.56(3)	104.91(3)
T / K	293(2)	273(2)
V / nm^3	1.412 9(5)	1.634 8(6)
Z	4	4
$D_c / (g \cdot cm^{-3})$	1.266	1.784
μ / mm^{-1}	0.088	1.269
$F(000)$	568	880
θ range for data collection / $(^\circ)$	3.06~27.46	1.00~27.48
Reflns collected	13 154	15 192
Unique reflns	3 217	3 752
R_{int}	0.059 5	0.040 1
GOF	1.046	1.112
Data with $I > 2\sigma(I)$, parameters	1 883, 193	2 582, 227
R_1^a, wR_2^b $I > 2\sigma(I)$	0.059 8, 0.113 3	0.035 8, 0.064 0
R_1^a, wR_2^b (all data)	0.112 3, 0.142 5	0.067 0, 0.099 1

^a $R_1 = \sum (|F_o| - |F_c|) / \sum |F_o|$; ^b $wR_2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]^{1/2}$, $w = [\sigma^2(F_o^2) + (AP)^2 + BP]^{-1}$ with $P = (F_o^2 + 2F_c^2)/3$, $A = 0.039\ 8$, $B = 0.436\ 6$ for L; $A = 0.010\ 8$, $B = 5.848\ 2$ for **1**

Table 2 Selected bond lengths (nm) and angles ($^\circ$) for L and **1**

L					
O1-C8	0.136 2(2)	O1-C16	0.142 7(3)	O2-C10	0.135 4(2)
O2-C7	0.140 8(2)	O3-C10	0.196(2)	N1-C15	0.132 6(3)
C8-O1-C16	117.4(2)	C10-O2-C7	117.9(2)	C15-N1-C12	116.8(2)
C1B/C1A-C2A-C3B/C3A	125.9(5)	C2A-C3B/C3A-C4	116.4(3)	C5-C4-C3B/C3A	122.2(2)

Continued Table 2

C9-C4-C3B/C3A	119.1(2)	N1-C12-C11	124.3(2)	N1-C15-C14	123.8(2)
O1-C8-C9	125.3(2)	O1-C8-C7	115.9(2)	O3-C10-C11	125.3(2)
1					
Ag-N1#1	0.225 1(4)	Ag-C1	0.237 7(5)	Ag-C2	0.244 0(5)
Ag-O6	0.248 3(4)	Ag-O4#2	0.277 0(4)	Ag-O6#2	0.269 0(5)
N1#1-Ag-C1	114.4(2)	N1#1-Ag-C2	139.1(1)	C1-Ag-C2	32.0(2)
N1#1-Ag-O6	116.6(1)	C1-Ag-O6	118.3(2)	C2-Ag-O6	86.4(2)
N1#1-Ag-O4#2	120.6(1)	N1#1-Ag-O6#2	88.6(1)	O4#2-Ag-O4#2	46.1(1)

Symmetry transformations used to generate equivalent atoms: #1: $-x+1, y+1/2, -z+3/2$; #2: $-x-1, y+1/2, -z+3/2$

2 Results and discussion

2.1 Crystal structures

The molecular structure of compound **L** is illustrated in Fig.1. **L** crystallizes in the monoclinic space group $P2_1/n$ and the mean planes through the benzyl and pyridine units make a dihedral angle of $75.6(1)^\circ$.

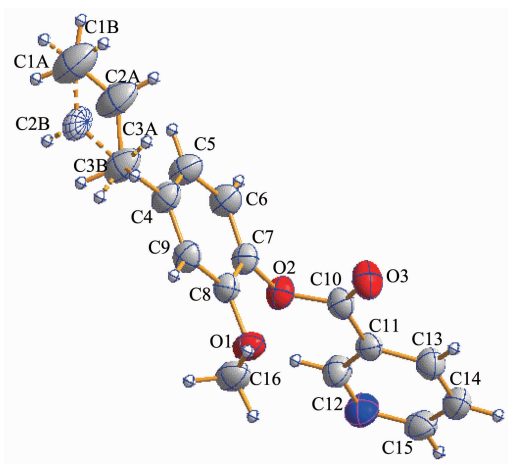


Fig.1 ORTEP view of compound **L** with ellipsoids at 35% probability levels

The association of molecules to form crystals is effected by the C–H \cdots O interactions (documented in Table 3). In this structure, there appear to be three types of C–H \cdots O interactions. The first type is C2 \cdots H2AA \cdots O1#1 hydrogen bond interaction to form a dimer (Fig.2a). The second type is to form a 2D layer structure involving C1–H1AB \cdots O2#2 hydrogen bonding interactions at the ends of the dimer (Fig. 2b). The third type also is a 2D layer (Fig.2c) but involves two modes of C–H \cdots O interactions, C6–H6A \cdots O3#3 as well as C14–H14A(H15A) \cdots O3#4 and C15–H15A \cdots O3#4, so one molecule links four different molecules through the C–H \cdots O3 interactions and at last forming a 3D supramolecular structure (Fig.2d).

The structure of compound **1** is shown in Fig.3. The Ag(I) atom is four-coordinated by one oxygen (O6) of nitrate anion, two carbon atoms (C1,C2) and one nitrogen (N1) from different ligands. The mean planes through the benzyl and pyridine units make a dihedral angle of $74.9(1)^\circ$, which is smaller than **L**.

The Ag–N1 bond distance is 0.225 4(3) nm, which

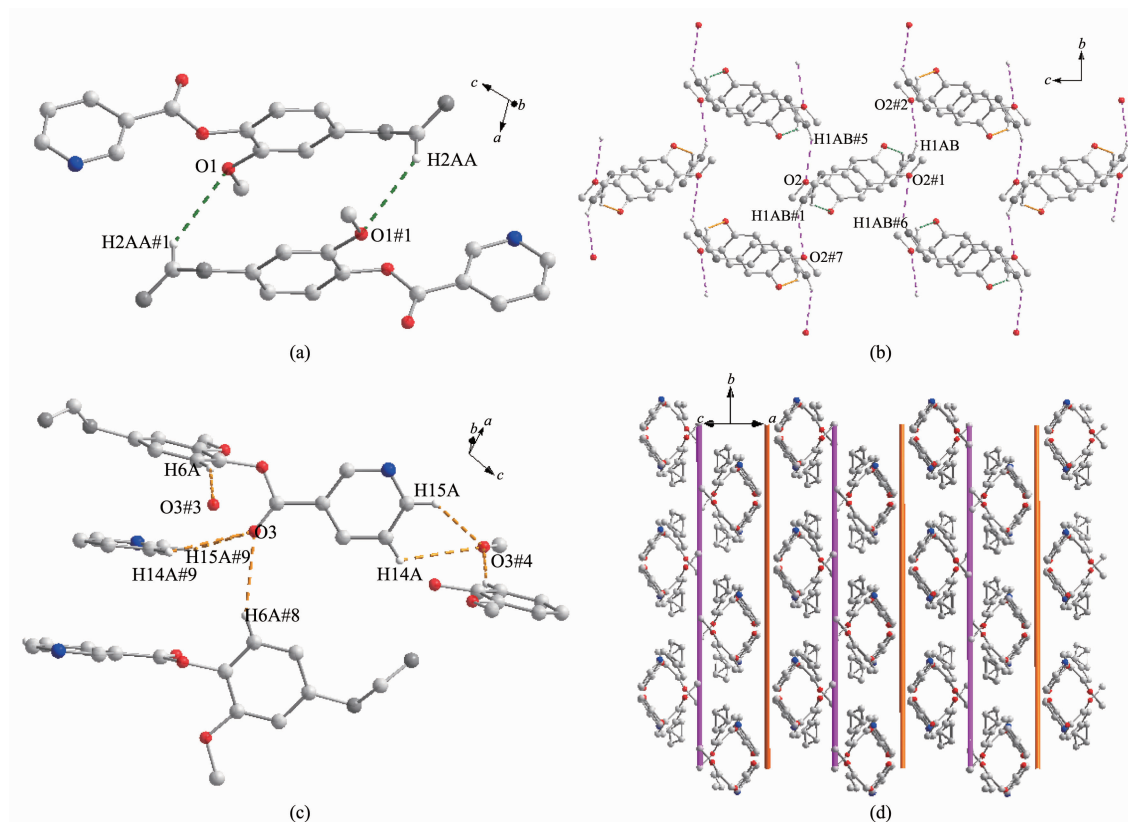
Table 3 C–H \cdots O hydrogen bonds for **L** and **1**

D–H \cdots A	$d(\text{D–H}) / \text{nm}$	$d(\text{H}\cdots\text{A}) / \text{nm}$	$d(\text{D}\cdots\text{A}) / \text{nm}$	$\angle \text{DHA} / (^\circ)$
L				
C2–H2AA \cdots O1#1	0.093 00(58)	0.279 8(2)	0.355 1(6)	138.8(4)
C1–H1AB \cdots O2#2	0.092 96(38)	0.287 9(2)	0.370 0(4)	148.0(2)
C6–H6A \cdots O3#3	0.092 99(23)	0.271 3(2)	0.357 0(3)	153.6(1)
C14–H14A \cdots O3#4	0.093 02(27)	0.287 2(2)	0.340 7(3)	117.8(2)
C15–H15A \cdots O3#4	0.092 92(21)	0.267 3(2)	0.332 4(3)	127.8(2)
Compound 1				
C1–H1A \cdots O4#2	0.097 02(48)	0.292 7(5)	0.325 6(8)	101.1(4)
C2–H2A \cdots O4#3	0.098 05(41)	0.294 8(5)	0.334 0(7)	105.0(3)

Continued Table 3

C3-H3A...O4#3	0.097 02(47)	0.298 7(5)	0.358 4(6)	120.9(3)
C5-H5A...O5#3	0.093 04(42)	0.296 2(5)	0.384 6(6)	159.1(3)
C8-H8A...O2#4	0.092 97(44)	0.277 6(3)	0.348 2(5)	133.6(3)
C10-H10A...O1#5	0.096 01(68)	0.267 8(3)	0.354 2(8)	149.9(4)
C10-H10B...O5#6	0.096 00(54)	0.283 5(6)	0.367 1(8)	146.0(4)
C10-H10C...O3#7	0.095 95(62)	0.275 3(3)	0.333 0(6)	119.4(4)
C14-H14A...O4#8	0.093 07(46)	0.292 2(5)	0.349 2(7)	120.8(3)
C14-H14A...O6#9	0.093 07(46)	0.266 8(4)	0.334 4(6)	130.1(3)
C15-H15A...O4#10	0.093 06(51)	0.282 2(4)	0.342 4(7)	123.4(3)
C16-H16A...O1#11	0.093 07(43)	0.271 4(3)	0.355 0(6)	149.9(3)
C16-H16A...O3#11	0.093 07(43)	0.265 5(3)	0.324 9(5)	122.4(3)

Symmetry codes: #1: $-x+1, -y+2, -z$; #2: $x-1/2, -y+5/2, z-1/2$; #3: $-x+1/2, y+1/2, -z+1/2$; #4: $x+1/2, -y+3/2, z+1/2$ for **L**; #2: $-x-1, y+1/2, -z+3/2$; #3: $x, -y+1/2, z-1/2$; #4: $-x, y+1/2, -z+3/2$; #5: $-x, -y, -z+1$; #6: $x, -y+1/2, z-1/2$; #7: $x, y-1, z$; #8: $-x, y+1/2, -z+3/2$; #9: $x+1, y, z$; #10: $-x, y+1/2, -z+3/2$; #11: $-x, 1-y, 1-z$ for **1**.

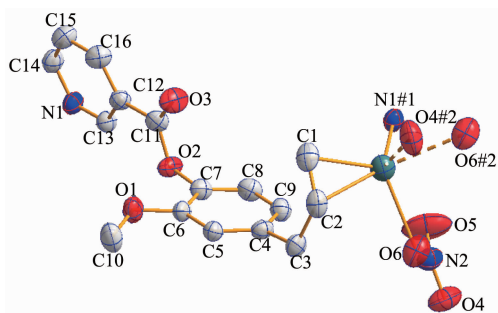


Only the hydrogen atoms involved in supramolecular interactions are shown for clarity; Symmetry code: #1: $-x+1, -y+2, -z$; #2: $x-1/2, -y+5/2, z-1/2$; #3: $-x+1/2, y+1/2, -z+1/2$; #4: $x+1/2, -y+3/2, z+1/2$; #5: $x+1/2, -y+5/2, z+1/2$; #6: $-x+1/2, y-1/2, -z-1/2$; #7: $-x+3/2, y-1/2, -z+1/2$; #8: $-x+1/2, y-1/2, -z-1/2$; #9: $x-1/2, -y+3/2, z-1/2$

Fig.2 (a) Dimer stepped motif formed with the C2-H2AA...O1 hydrogen bonding interaction; (b) 2D layers formed by the C1-H1AB...O2 hydrogen bonding interaction(the pyridine rings were omitted for clarity); (c) 2D layers formed by the C-H...O3 hydrogen bonding interaction; (d) View of the 3D structure of **L**

is comparable to those previously reported Ag(I)-N_{py} coordination distances^[16-18]. The silver ion is coordinated by the C1-C2 double bond of one molecule with Ag-C

bond distances ranging from 0.237 9(4) nm to 0.244 3(4) nm. The Ag-C distance is similar to those of the reported silver complexes with alkenes and arenes^[19-21],



Hydrogen atoms are omitted for clarity; Symmetry codes: #1: $-x+1, y+1/2, -z+3/2$; #2: $-x-1, y+1/2, -z+3/2$

Fig.3 ORTEP view of coordination environment of Ag ions and atomic labeling in **1** with ellipsoids at 35% probability levels

whereas the C1-C2 double bond is consistent with the IR spectrum which suggests the C=C stretching vibrations of the ligand. The elongation of olefinic bond is mainly observed in the basal ligands with the C1-C2 distance of 0.134 3(5) nm.

The Ag...O4#2 (O6#2) distance of 0.268 8(3) (0.277 4(3)) nm is shorter than the van der Waals contact distance of 0.324 nm. Therefore, the Ag...O distances suggest non-negligible interactions between Ag(I) and NO₃⁻ group, which may be described as a semi-coordination mode. Hence Ag(I) can be regarded as a pseudo-six-coordinated environment (Fig.3). The L ligand connects the Ag(I) ions together by N atoms and allyl groups to generate a 1D left-handed helical chain of [AgL]_∞ around the crystallographic 2₁ screw axis with the pitch of 0.698 6(1) nm along the *b* axis. And the NO₃⁻ group connects Ag(I) ions of adjacent chains together by the Ag...O weak interactions to generate a 1D right-handed helical chain of [AgNO₃]_∞ around the crystallographic 2₁ screw axis with the

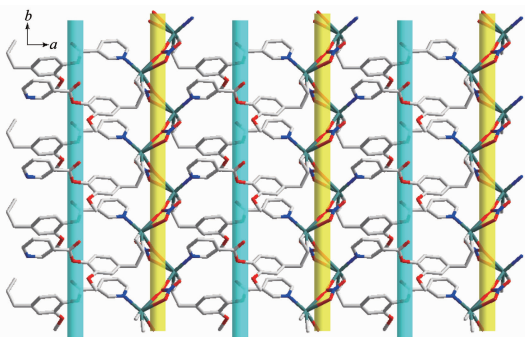


Fig.4 View of two-dimensional layer comprising the right (yellow)- and left (green)-handed helical chains

pitch of 0.698 6(1) nm along the *b* axis. Thus two kinds of alternated helices are connected in ...ABAB... fashion to form a 2D achiral layer (Fig.4).

The resulted 2D layers are further connected to form the 3D supramolecular architecture due to weak C-H...O hydrogen bonds (Table 3).

2.2 Fluorescence properties

The photoluminescence properties of compounds L and **1** were studied in the solid state at room temperature. As shown in Fig.5, L and **1** show the similar emission and excitation spectra, which exhibit luminescence in violet region with centers at $\lambda_{\text{max}}=364$ nm ($\lambda_{\text{ex}}=334$ nm). These emissions can be attributed to neither metal-to-ligand charge transfer (MLCT) nor ligand-to-metal charge transfer (LMCT) because the Ag(I) ion has *d*¹⁰ configuration with one positive charges. The d-orbitals are contracted and therefore the electrons in these orbitals are much less accessible for back bonding to p-acceptor ligands, and silver cation has a weak electro accepting nature with respect to electrons from L. So the luminescent emissions of **1** is from the ligand, which can be ascribed to the intraligand $\pi^*-\pi$ transitions^[10,22-23].

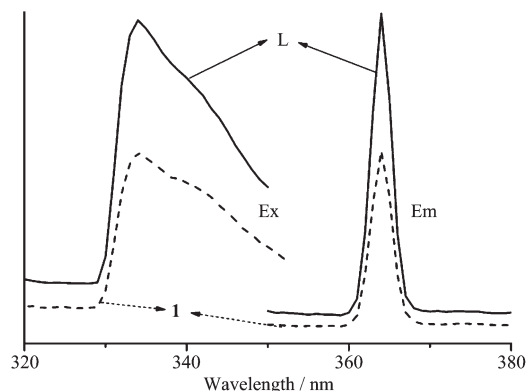


Fig.5 Emission and excitation spectra for compounds L and **1**

3 Conclusions

In summary, Silver-containing complex **1** was synthesized through the interactions of 4-allyl-2-methoxyphenyl nicotinate (L) and silver nitrate. Two new compounds L and **1** were confirmed by single crystal X-ray diffraction study.

The observations of the luminescent properties

indicate that the two complexes showed violet-light photoluminescent, but **1** may be excellent candidate of photoactive materials, which are thermally stable and insoluble in common organic solvents.

Acknowledgements: The authors would like to thank Prof. Dr. ZHENG Yue-Qing for initiating our interest in this work and Dr. XU Wei and ZHU Hong-Lin for their comments on the manuscript. Acknowledgement is made to the classmates WAN Dan-Weng, WEN Gang-Zhu et al. for the helps and X-ray diffraction measurements.

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