

Synthesis, structure and spectroscopic properties of a novel compound *bis*(benzylamino)silver(I) benzylcarbamate

Jianguo Liu,^{a*} Xiaoyan Zeng^a and Hua Li^b

^aLaser and Terahertz Functional Laboratory, Wuhan National Laboratory for Optoelectronics, Huazhong University of Science and Technology, Wuhan 430074, People's Republic of China, and ^bCollege of Chemistry and Molecular Science, Wuhan University, Wuhan 430072, People's Republic of China

Correspondence e-mail: ljg712@yahoo.com.cn

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A novel silver-containing compound, *bis*(benzylamino)silver(I) benzylcarbamate, with an unusual molecular structure is easily synthesized by the reaction of benzylammonium benzylcarbamate and silver oxide. It crystallizes in the triclinic crystal system with the space group $P\bar{1}$ with $a = 5.2006$ (5), $b = 14.6298$ (15), $c = 14.7246$ (15) Å, $\alpha = 68.729$ (2), $\beta = 83.507$ (2), $\gamma = 85.412$ (2)° and $Z = 2$. In the crystal, one Ag atom coordinates with the two amino groups in two benzylamine molecules, and there are no silver–silver and silver–oxygen interactions. The carboxylate groups take part in balancing the electric charge and forming hydrogen bonds. Both the compound and the starting material benzylammonium benzylcarbamate exhibit room-temperature solid-state emissions with the peaks at 300 and 406 nm, respectively.

1. Introduction

A number of silver-containing compounds including perfluorinated carboxylate silver(I) (Zheng *et al.*, 2001), perfluorinated carboxylate trimethylphosphine silver(I) (Szyk *et al.*, 2001; Chi *et al.*, 1996), (β -diketanato) silver(I) bis(trimethylsilyl)acetylene (Chi & Lu, 2001), silver(I) alkanoates (Binnemans *et al.*, 2004; Jaber *et al.*, 1997), bis-triphenylphosphine-silver(I) stearate (Whitcomb & Rogers, 1996), silver(I) β -diketiminato complexes (Chiong & Daugulis, 2006), crown ether silver(I) carboxylate complex (Ferguson *et al.*, 1984), and silver neodecanoate have been synthesized and characterized because of their wide applications as a catalyst, bactericide, precursor of chemical vapor deposition (CVD), illuminated semiconductor material *etc.* Thus, great progress has been made in this area.

As is well known, aliphatic amines or ammonia are able to coordinate with silver(I) ions. For example, the reactions of aliphatic amines with silver(I) ions result in silver–amine complexes. However, the complexes are unstable and prone to decomposition and yielding metallic silver (Emeleus *et al.*, 1973; Cotton & Wilkinson, 1962).

It had been reported (Zheng *et al.*, 2006) that silver(I) oxide was able to react with amines plus lactones (or lactams, carbonic esters and *cyclo*-anhydrides) to produce silver metal-organic compounds. The compounds are highly soluble in water and able to decompose thoroughly at 523–573 K. However, the authors neither characterized the compounds nor described their structures. By following their work, we found the novel silver-containing compound *bis*(benzylamino)silver(I) benzylcarbamate. It has been shown that the compound has an unusual molecular structure, which is soluble in water and/or common organic solvents and able to decompose thoroughly at lower temperatures (below 473 K).

Table 1

Crystal data and structure refinement summary for $C_{22}H_{26}N_3O_2Ag$ (CCDC deposition 804544).

Crystal data	
Chemical formula	$C_{22}H_{26}AgN_3O_2$
M_r	472.33
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	293
a, b, c (Å)	5.2006 (5), 14.6298 (15), 14.7246 (15)
α, β, γ (°)	68.729 (2), 83.507 (2), 85.412 (2)
V (Å ³)	1036.37 (18)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	0.99
Crystal size (mm)	0.32 × 0.28 × 0.26
Data collection	
Diffractometer	Bruker
Absorption correction	Multi-scan <i>SADABS</i>
T_{min}, T_{max}	0.741, 0.782
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6094, 4009, 3622
R_{int}	0.017
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.027, 0.068, 1.07
No. of reflections	4009
No. of parameters	254
No. of restraints	0
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	0.33, -0.51

Computer programs used: *SMART*, *SAINT* (Bruker, 2009), *SHELXS97*, *SHELXL97* (Sheldrick, 2008).

Furthermore, it has a fluorescence property. Now it has been applied in high-performance thick-film paste with low-temperature sintering (Liu *et al.*, 2010). It is expected that it has greater potential to apply in electrically conductive ink, electrically conductive adhesive, organic light-emitting diode (OLED) and so on (Teng & Vest, 1988; Jung *et al.*, 2007; Kamyshny *et al.*, 2005).

2. Experimental

2.1. Materials

Chemicals including absolute methanol (CH₃OH), benzylamine (C₆H₅CH₂NH₂), silver oxide (Ag₂O), ethanol (CH₃CH₂OH), ether ((CH₃CH₂)₂O), ethyl acetate (CH₃COOC₂H₅), chloroform (CH₃Cl), dimethyl sulfoxide (CH₃SOCH₃) and acetone (CH₃COCH₃) were analytical reagent (AR)-grade, obtained from Sinopharm Chemical Reagent, and used as received without further purification. Dry carbon dioxide (CO₂) had the purity of 99.99%.

2.2. Synthesis of bis(benzylamino)silver(I) benzylcarbamate

First benzylammonium benzylcarbamate (C₆H₅CH₂NHCOO⁻NH₃⁺CH₂C₆H₅) was prepared according to George & Weiss (2001), Wright & Moore (1948), Katchalski *et al.* (1951) and Hoerr *et al.* (1944).

Next, at room temperature and with stirring, silver oxide (2.5 g, 0.011 mol) was added into a methanol solution of

benzylammonium benzylcarbamate (10.32 g, 0.04 mol) and the reaction was left for 3 h. The solution was then filtered through a 0.25 μm Teflon filter, and a colorless, transparent and clear filtrate was obtained. After the solvent methanol was removed under vacuum, the crude product of bis(benzylamino)silver(I) benzylcarbamate was obtained. The crude product was further purified twice by recrystallization from 1:1 (volume-to-volume) methanol and ether. The yield was 82% (7.74 g); m.p. 383–385 K. FT-IR (KBr pellets, cm⁻¹): 3445, 3296, 3244 (NH₂, NH), 3085, 3063, 3026, 1588, 1492, 1452, 746, 695 (C₆H₅), 2946–2819, 1470, 729 (CH₂), 1642 (O=C–O). ¹H NMR (400 MHz, CD₃SOCD₃) δ: 3.75 (s, 6H, 3 × CH₂), 4.09 (d, 4H, 2 × NH₂), 5.86 (s, 1H, NH), 7.14–7.34 (m, 15H, 3 × C₆H₅). ¹³C NMR (100 MHz, CD₃OD) δ: 164.10, 164.07, 141.23, 136.45, 128.61, 128.19, 128.06, 127.95, 126.84, 126.34, 45.18, 43.57. MS (ESI(+)): m/z 365.2 [$M + H$]⁺, 367.2 [$M + 2 + H$]⁺. Anal.: calc. for C₂₂H₂₆N₃O₂Ag: C 55.94, H 5.55, N 8.90; found: C 55.92, H 5.58, N 8.96.

2.3. Measurements and characterization

FT-IR, ¹H and ¹³C NMR, and MS (mass spectrometry) were carried out using an EQUINOX 55 FT-IR spectrophotometer from KBr (potassium bromide) pellets, a Mercury VX-300 NMR spectrometer with tetramethylsilane (TMS) as internal standard, and an Agilent 1100LC/MSD (liquid chromatography/mass selective detector) system. Elemental analysis was determined on a Vario EL III elemental analyzer (Elementar Corporation, Germany) with acetanilide as the standard. Thermal analyses were performed using a Perkin-Elmer DSC-7 and TGA-7 thermal analyzer (Perkin-Elmer Inc., USA) by differential thermal analysis (DTA) and thermogravimetric analysis (TGA) under nitrogen at a heating rate of 10 K min⁻¹. UV–vis absorption and emission spectra of the solution and solid were recorded on a Lambda 35 UV–vis spectrophotometer (Perkin-Elmer Inc., USA) and a FP-6500 fluorescence spectrophotometer.

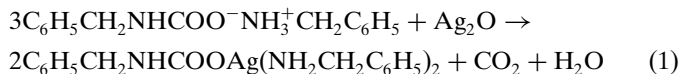
At room temperature and by slow isothermal evaporation, the X-ray quality crystal of bis(benzylamino)silver(I) benzylcarbamate was grown from a solution in methanol:ether = 1:1 and a single crystal of 0.32 × 0.28 × 0.26 mm³ in size was selected for X-ray analysis. Crystallographic data were collected on a Bruker Smart Apex II X-ray single-crystal diffractometer (Bruker Corporation, Germany). The structure was solved using the *SHELXS97* program (Sheldrick, 2008) and refined using *SHELXL97* (Sheldrick, 2008). All non-H atoms were refined anisotropically. All H atoms were introduced in their idealized sites and refined using a rigid model. Detailed crystal data and structural refinement parameters for C₂₂H₂₆N₃O₂Ag are summarized in Table 1.

3. Results and discussion

3.1. Synthesis and structure of bis(benzylamino)silver(I) benzylcarbamate

Experiment shows that bis(benzylamino)silver(I) benzylcarbamate can be easily prepared by the reaction of

benzylammonium benzylcarbamate with silver oxide. There is a gas discharge during the reaction. We infer that the reaction may proceed according to the following equation. However, the detailed reaction mechanism needs to be further investigated later.



Bis(benzylamino)silver(I) benzylcarbamate is a white crystalline powder, which slowly turns fuscous on exposure to daylight. It is soluble in such solvents as water, methanol, ethanol, ether, ethyl acetate, chloroform, dimethyl sulfoxide and acetone. In addition, its aqueous or methanol solution is very stable and can be stored without color change for several months in daylight. As is well known, many silver-containing compounds (*e.g.* silver oxide, silver carbonate, silver halide, silver salts of primary and secondary fatty acid) are quite insoluble both in water and common organic solvents (Whitcomb & Rogers, 1996; Nao *et al.*, 2007; Houg *et al.*, 2006), except silver nitrate. Although silver salts of tertiary fatty acids (*e.g.* silver neodecanoate) have good solubility in organic solvents, the solutions obtained are light-sensitive (Nao *et al.*, 2007; Houg *et al.*, 2006; Chun *et al.*, 2009). Therefore, bis(benzylamino)silver(I) benzylcarbamate has some advantages.

In the TGA and DTA curves of bis(benzylamino)silver(I) benzylcarbamate (Fig. 1), it can be seen that the compound, which is thermally stable below 353 K, starts to decompose at higher temperatures. The decomposition is completed around 443 K. Here, the corresponding weight ratio in the TGA curve is approximately 22.70 wt %, which is assigned to the content of silver in the compound. In the DTA curve the endothermic peak at 384.2 K corresponds to the melting point of the compound. It is a good fit with the value (383–385 K) measured by the capillary method. The endothermic decomposition peak is at 426.3 K.

According to the results of elemental and thermal analyses, it can be inferred that bis(benzylamino)silver(I) benzylcar-

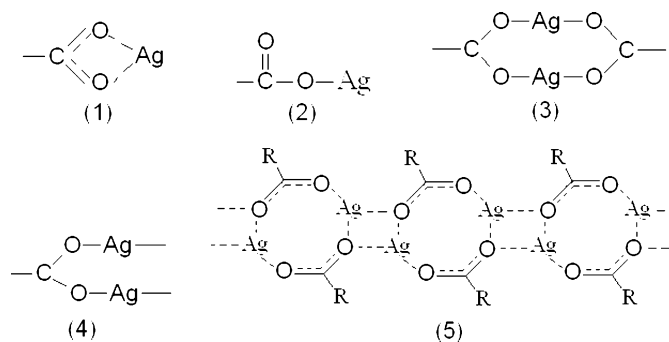


Figure 2
Examples of the structures containing Ag–O interactions.

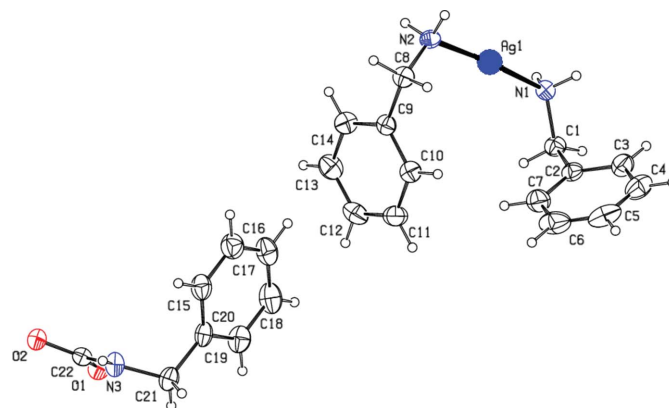


Figure 3
ORTEP diagram of the molecular structure of bis(benzylamino)silver(I) benzylcarbamate.

bamate has the empirical formula $\text{C}_{22}\text{H}_{26}\text{N}_3\text{O}_2\text{Ag}$. MS analysis reveals that the compound has the formula weight 472 (*i.e.* $365 - 1 + 108 = 472$ or $367 - 2 - 1 + 108 = 472$), so it has the molecular formula $\text{C}_{22}\text{H}_{26}\text{N}_3\text{O}_2\text{Ag}$.

A comprehensive deduction based on FT-IR (Fourier transform IR spectrometry), ^1H NMR and ^{13}C NMR analyses indicates that the compound has such functional groups as C_6H_5 , CH_2 , NH_2 , NH and $\text{O}-\text{C}=\text{O}$. No peaks at 1730, 1690, 1675 and 1680 cm^{-1} in its FT-IR spectrum show that the compound does not contain the structures of the Ag–O interaction listed in Fig. 2, which often appear in the structures of long straight-chain alkyl carboxylate silver (Szyk *et al.*, 2001; Alcock *et al.*, 1976).

X-ray single-crystal diffraction analysis demonstrates that the compound has the molecular structure (Fig. 3) where the Ag atom is two-coordinately bonded to two amino groups from two different benzylamine molecules. Furthermore, the three atoms of N1, Ag and N2 are almost linear [*i.e.* the bond angle of $\text{N}2-\text{Ag}-\text{N}1$ is $175.96(7)^\circ$, see supplementary material¹]. Unexpectedly, the two O atoms from the carboxylate group do not coordinate with the Ag atom, and no

¹ Supplementary data for this paper are available from the IUCr electronic archives (Reference: BP5042). Services for accessing these data are described at the back of the journal.

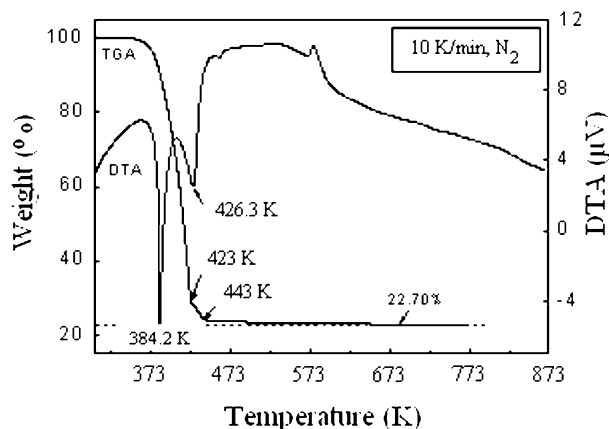


Figure 1
TGA and DTA curves of bis(benzylamino)silver(I) benzylcarbamate.

Table 2

Hydrogen-bonding geometry (Å, °) for C₂₂H₂₆N₃O₂Ag.

<i>D</i> –H··· <i>A</i>	<i>d</i> (<i>D</i> –H)	<i>D</i> (H··· <i>A</i>)	<i>d</i> (<i>D</i> ··· <i>A</i>)	∠ (<i>DHA</i>)
N3–H3A···O1 ⁱ	0.86	2.29	3.087 (2)	153.6
N2–H2B···O1 ⁱⁱ	0.90	2.10	2.931 (2)	154.0
N2–H2A···O2 ⁱⁱⁱ	0.90	2.12	2.943 (2)	151.3
N1–H1D···O1 ^{iv}	0.90	2.12	3.011 (2)	170.5
N1–H1C···O2 ^v	0.90	2.32	3.037 (2)	136.2

Symmetry codes: (i) *x* + 1, *y*, *z*; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x + 2, -y + 1, -z + 1$; (iv) *x*, *y* + 1, *z* – 1; (v) *x* – 1, *y* + 1, *z* – 1.

Ag–O interaction is present in the structure. Thus, the compound cannot contain a one-dimensional chain structure and two-dimensional layer structure (Binnemans *et al.*, 2004; Jaber *et al.*, 1994, 1997; Whitcomb & Rogers, 1996; Alcock *et al.*, 1976). The carboxylate group balances the charge of the compound. This further supports the above conclusion from the FT-IR spectrum. So it can be said that the compound has an unusual molecular structure differing from that of other carboxylate-containing silver compounds, in which the carboxylate group usually interacts (*e.g.* coordination effect) with the Ag atom (Szyk *et al.*, 2001; Binnemans *et al.*, 2004; Jaber *et al.*, 1994, 1997; Whitcomb & Rogers, 1996; Alcock *et al.*, 1976; Jaber *et al.*). For example, in the molecular structure of bis-triphenylphosphine-silver(I) stearate [CH₃(CH₂)₁₆COOAg(P(C₆H₅)₃)₂], the Ag atom is four-coordinate: the carboxylate group is symmetrically bonded to the Ag atom while the triphenylphosphine ligands occupy the third and fourth coordination sites (Whitcomb & Rogers, 1996). The difference between these two kinds of molecular structures may be due to the variation of the functional groups that connect directly with the carboxylate, *i.e.* one connects with the alkyl or aralkyl group, and the other links with the amino group.

The compound crystallizes in a triclinic crystal system and space group *P* $\bar{1}$ with *a* = 5.2006 (5), *b* = 14.6298 (15), *c* = 14.7246 (15) Å, α = 68.729 (2), β = 83.507 (2), γ = 85.412 (2)° and *Z* = 2 (Table 1). The starting material benzylammonium benzylcarbamate also crystallizes in the same crystal system and space group, but its unit-cell dimensions are *a* = 5.8051 (3), *b* = 9.4469 (5), *c* = 25.2290 (10) Å, α = 79.301 (7), β = 89.010 (6), γ = 89.649 (7)° and *Z* = 4 (Tiritiris & Kantlehner, 2011). The contents of the unit cell (Fig. 4) of bis(benzylamino)silver(I) benzylcarbamate make a hexahedron structure, in which seven Ag atoms are present. Three of them occupy the three crucial nodes of the hexahedron, and the other four locate at the fourth. However, there is no interaction between the Ag atoms.

Characteristically, it is observed that a large quantity of hydrogen bonds is formed in the crystal structure (Table 2). These hydrogen bonds can be classified into two categories. One is formed between the H atom attached to the N atom from the benzylcarbamate group and the O atom in another adjacent benzylcarbamate group (bond length 2.29 Å), and the other is between the H atom attached to the N atom of the benzylamine molecule and the O atom (the bond length is from 2.10 to 2.32 Å). It is also found that in the hydrogen bond

of the second category, two H atoms attached to the same N atom form hydrogen bonds with two O atoms from two different carboxylate groups. Thus, each O atom involves two hydrogen bonds. Among the atoms involved in hydrogen bonding, no eight-membered ring is formed, which usually exists in the crystal structures of ammonium carbamates (Tiritiris & Kantlehner, 2011; Tiritiris *et al.*, 2011).

In Fig. 4 it is obvious that there are no such interactions as the valence bonds, Coulomb forces and hydrogen bonds between the atoms and molecules from different nodes. So it can be inferred that weak interactions such as van der Waals interactions should exist in the crystal structure. The hydrogen bond and covalence bond, together with the van der Waals interactions, sustain the stability of the three-dimensional crystal structure.

3.2. Spectroscopic properties of bis(benzylamino)silver(I) benzylcarbamate

Inorganic organic hybrid coordination compounds, especially with *d*¹⁰ metal centers, have been investigated for fluorescence properties due to their potential applications as luminescent materials such as light-emitting diodes (LEDs), but they usually need complicated procedures for preparation and purification (Tang & Vanslyke, 1987; Li *et al.*, 2011). On the other hand, bis(benzylamino)silver(I) benzylcarbamate can easily be prepared and purified, so its spectroscopic properties have been explored.

The UV absorption spectrum of bis(benzylamino)silver(I) benzylcarbamate (curve 3) in methanol solution is shown in Fig. 5. Two absorption peaks can be seen at 258 and 219 nm, respectively, which are the same as the UV absorption peaks of the ligand benzylamine (curve 1) and the reactant benzylammonium benzylcarbamate (curve 2). They can be assigned to the UV absorption correlating to the benzene ring and the

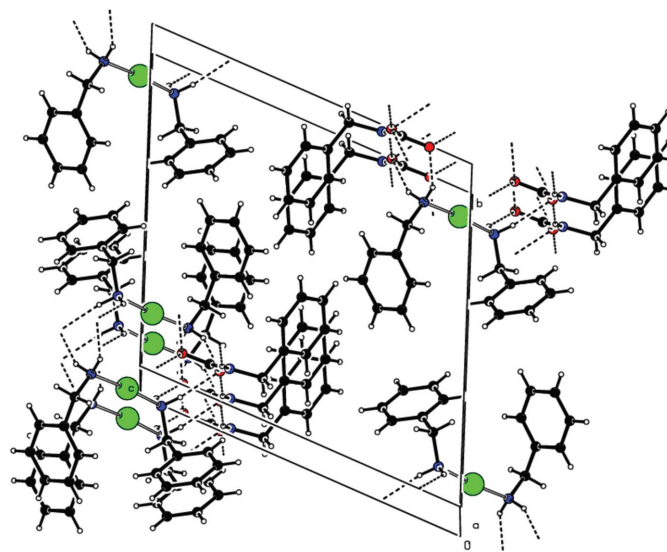


Figure 4
The packing arrangement in the unit cell of bis(benzylamino)silver(I) benzylcarbamate.

amino group, respectively. However, in their ether solution (inset in Fig. 5) the absorption peak at the shorter wavelength of 219 nm red-shifts to 233 nm. This may be related to the aprotic solvent of ether. Additionally, all the three compounds have no apparent absorption in the visible range from 400 to 800 nm.

Experiment shows that the crystal of bis(benzylamino)-silver(I) benzylcarbamate is able to emit blue-purple light when it is irradiated by UV light. In the room-temperature

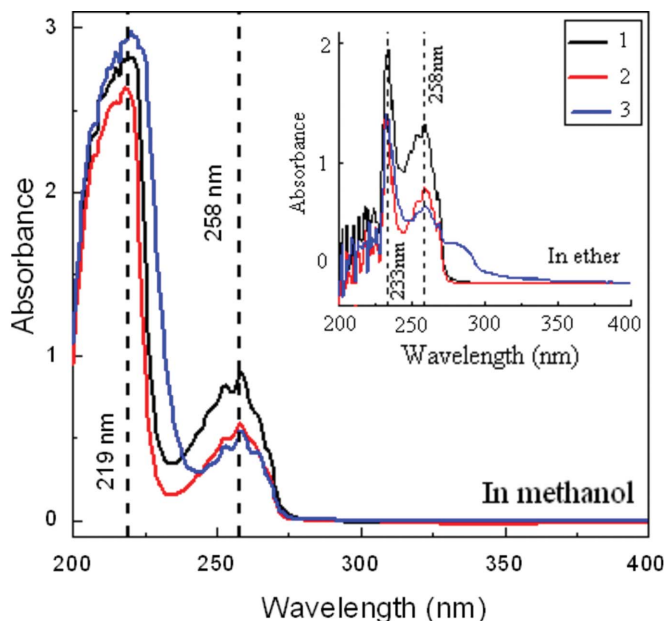


Figure 5 Room-temperature UV-vis absorption spectra of benzylamine (1), benzylammonium benzylcarbamate (2) and bis(benzylamino)silver(I) benzylcarbamate (3) in methanol and in ether (inset), with a concentration of 0.4 mg ml^{-1} .

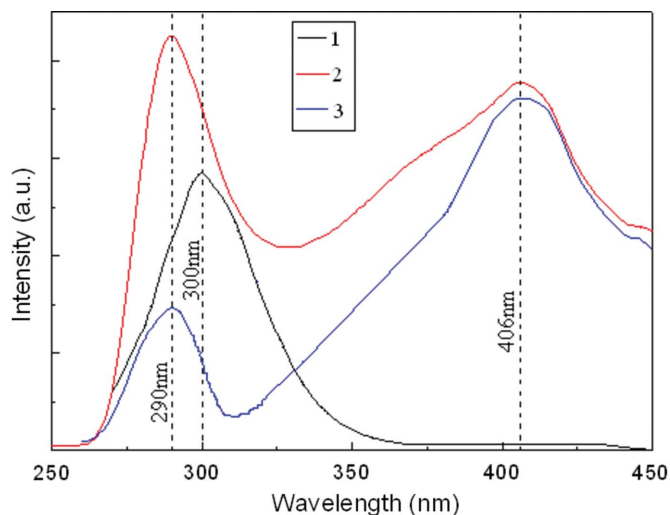


Figure 6 Room-temperature fluorescence spectra of liquid benzylamine (1), solid benzylammonium benzylcarbamate (2) and solid bis(benzylamino)silver(I) benzylcarbamate (3) with excitation at 255, 240 and 240 nm, respectively.

fluorescence spectrum of solid bis(benzylamino)silver(I) benzylcarbamate (Fig. 6 curve 3), it is found that it has two emission peaks at 290 and 406 nm, respectively. Also, solid benzylammonium benzylcarbamate (curve 2) has these two emission peaks, but the peak at 290 nm is more intense. While in the fluorescence spectra of liquid benzylamine (curve 1), only one peak at 300 nm appears, which infers that the emission peak of bis(benzylamino)silver(I) benzylcarbamate at 290 nm may be related to the ligand benzylamine. However, the attribution of the emission peak at 406 nm needs to be further explored in the near future.

4. Conclusion

A novel compound bis(benzylamino)silver(I) benzylcarbamate has been synthesized and characterized. It has the molecular formula $\text{C}_{22}\text{H}_{26}\text{N}_3\text{O}_2\text{Ag}$ and the structural formula $\text{C}_6\text{H}_5\text{CH}_2\text{NHCOOAg}(\text{NH}_2\text{CH}_2\text{C}_6\text{H}_5)_2$. In the crystal structure of the compound (triclinic crystal system and $P1$ space group) each Ag atom coordinates only with two amino groups from two benzylamine molecules, the carboxylate group balances electric charge and forms a hydrogen bond with the H atom at the N atom, and these hydrogen bonds participate in sustaining the stability of the three-dimensional structure. The UV absorption spectrum in methanol solution shows that both the compound and the starting material benzylammonium benzylcarbamate have two absorption peaks at 258 and 219 nm, and there is no apparent absorption in the visible range from 400 to 800 nm. They also exhibit room-temperature solid-state emission with the peaks at 290 and 406 nm, respectively.

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