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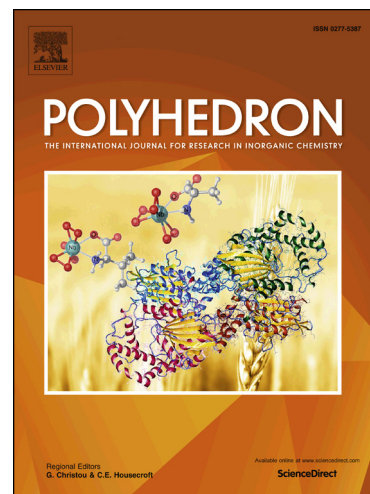
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# Synthesis and characterization of new silver(I) naphthalenedisulfonate complexes with heterocyclic N-donor ligands: packing analyses and antibacterial studies

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## Abstract

Four new silver(I) naphthalenedisulphonate complexes, [Ag<sub>2</sub>(1,5-nds) ( $\beta$ -Pic)<sub>4</sub>].2H<sub>2</sub>O (**1**), [Ag<sub>2</sub>(1,5-nds)( $\gamma$ -Pic)<sub>4</sub>].6H<sub>2</sub>O (**2**), [Ag<sub>2</sub>(2,6-nds) ( $\beta$ -Pic)<sub>4</sub>] (**3**) and [Ag<sub>2</sub>(2,6-nds) ( $\gamma$ -Pic)<sub>4</sub>].2H<sub>2</sub>O (**4**) have been synthesized using appropriate starting materials at ambient temperature and normal reaction conditions and fully characterized by spectroscopic methods (FT-IR, NMR). The structures of all the four complexes were determined using single crystal X-ray diffraction techniques and a detailed analysis of the role of weak non-covalent interactions in the solid state (hydrogen bonds, argentophylic interactions,  $\pi\cdots\pi$  interactions) were investigated. Antibacterial activities of all complexes have been evaluated, indicating their high efficacy against both gram positive and gram negative bacterial strains.

**Keywords:** Silver(I) complexes; naphthalenedisulfonate; spectroscopy; X-ray crystallography; antibacterial activity

## 1. INTRODUCTION

The germicidal properties of silver, due to the oxidation of the metal to the  $\text{Ag}^+$  ion, have been exploited since 1200 BC. The Greeks and Romans, for instance, used to keep water in silver vessels, while some Ag(I) salts, particularly silver nitrate, have been used since the medieval age for topical applications in the treatment of chronic wounds or ulcers.[1] A renewed interest in medical uses of silver salts [2] aroused after the second world war and is still continuing, also in view of a possible alternative to antibiotics to overcome the bacterial resistance problems. [3] Nowadays the research in this field is devoted to the synthesis of new, safer and bio-available silver compounds, utilized in numerous products and medical devices, where the silver can be present in metallic, ionic or nanoparticulate form. [4]

Though seemingly simple and conceptually well defined to occur, practical feasibility of the design and successful synthesis of a coordination complex with well conceived properties depend upon appropriate choice of the metal ion(s) and organic ligand(s). Silver(I) metal ion exhibits flexible coordination numbers and geometries because of its unique electronic configuration  $4d^{10} 5s^1$ , for example: linear (coordination number(CN) = 2), trigonal (CN=3), tetrahedral (CN=4), distorted pentagonal planar or trigonal bipyramidal (CN=5), octahedral (CN=6) *etc.* Moreover, belonging to the  $d^{10}$  class of metal ions, Ag(I) can be involved in the so-called ‘argentophilic’ Ag...Ag interactions, that are able to influence the coordination geometries possibly leading to the formation of dimers, oligomers, infinite chains, *etc.* [5] The complex properties can be also tuned by varying the number and type of ligands. In this context, it is also worth mentioning that many characteristic features (solubility, stability, colour change, melting point, spin crossover behavior, chirality transfer *etc.*) stem from structure directing non-covalent interactions (hydrogen bonding, halogen bonding, C-H... $\pi$  and  $\pi$ ... $\pi$  interactions *etc.*) which ultimately lead to the thermodynamic stability of the complex in the condensed phase. [6] As a result, such framework complexes (generated and stably sustained through supramolecular interactions) have been attracting extensive attention of chemists, especially in the field of material science due to their intriguing architectures and

potential applications in medicine, luminiscence, magnetism, catalysis, sensing, gas storage/separation and non-linear optics. [7-15]

In view of our interest in both antimicrobial properties and supramolecular assemblies of transition metal complexes, we report in this paper the synthesis of new  $\text{Ag}^+$  complexes, i.e.  $[\text{Ag}_2(1,5\text{-nds})(\beta\text{-Pic})_4]\cdot 2\text{H}_2\text{O}$  (**1**),  $[\text{Ag}_2(1,5\text{-nds})(\gamma\text{-Pic})_4]\cdot 6\text{H}_2\text{O}$  (**2**),  $[\text{Ag}_2(2,6\text{-nds})(\beta\text{-Pic})_4]$  (**3**) and  $[\text{Ag}_2(2,6\text{-nds})(\gamma\text{-Pic})_4]\cdot 2\text{H}_2\text{O}$  (**4**), using a synthetic supramolecular association strategy [16,17] employed to generate mixed ligand metal complexes. The antibacterial-efficacy of these newly synthesized silver(I) complexes and their starting materials, i.e. respective disodium salts, against gram positive and gram negative bacterial strains have also been tested.

We took the cue of using the picoline molecules from some recent publications on active silver complexes [18,21] in which N-aromatic ligands such as nicotinate derivatives, pyrazines or pyridylamine have been used, with the intent of investigating the possibility of maintaining some germicidal properties in spite of the less complexity of the ligands. As for the choice of naphthalene disulfonate dinegative ions, they have the role of maximizing the molecular association in the crystals giving origin to complex packing architectures.

## 2. EXPERIMENTAL

### 2.1. Materials and physical measurements

Analytical grade reagents were used throughout this work without any further purification. Two naphthalenedisulfonates used in this work have been purchased from Sigma-Aldrich Limited, India. All solvents used in the work were purchased from Fischer Scientific Limited, India.

Carbon, hydrogen and nitrogen were measured micro-analytically by an automatic Perkin Elmer 2400 CHN elemental analyzer and metal content (silver) in these hybrid inorganic-organic complexes were determined gravimetrically by standard literature methods [22]. FT-IR spectra were recorded with PERKIN ELMERSPECTRUM RXFT-IR system in solid state. Multinuclear NMR was recorded on BRUKER AVANCE II 400 MHz spectrophotometer.

## 2.2. Antibacterial activities

### 2.2.1 Agar well diffusion assay

Agar well diffusion assay was performed to evaluate the antibacterial activity of the four silver complexes and their starting material. Briefly, 90 mm sterile nutrient agar plates were prepared and incubated overnight at 37°C. Next day, log cultures of *Staphylococcus aureus*, *Klebsiella pneumoniae*, *Pseudomonas aeruginosa* and *Escherichia coli* having an optical density of 0.5 at 600 nm were spread plated on separate nutrient agar plates. Wells were punched and 50 µl of each silver complex and their starting material (40 mg/ml) was added to separate wells on each culture plate. Sterile distilled water was used as solvent control. Silver (I) complexes and disodium salts were allowed to diffuse in nutrient agar for 1-2 hours in a laminar hood and plates were transferred to an incubator at 37°C for 18-24 hours. Next day, plates were observed for zone of inhibition around the wells.

### 2.2.2 Minimum Inhibitory Concentration (MIC) and Minimum Bactericidal Concentration (MBC) determination

MIC of silver complexes was determined by microtitre plate broth dilution method. Briefly 100 µl of log culture of *S. aureus*, *K. pneumoniae*, *P. aeruginosa* and *E. coli* having optical density of 0.5 at 600 nm were added to separate wells of microtiter plate. 100 µl of varying concentrations of silver complexes and their starting materials i.e., respective disodium salts (80-0.15 mg/ml) suspended in sterile distilled water were added to each well containing bacterial cultures. The plate was incubated at 37°C for 24 hours and after incubation each well was observed for visual turbidity of culture. The lowest concentration showing no turbidity was considered as MIC of that complex. For determination of MBC, 100 µl of contents from each well were spread plated on nutrient agar plates and plates incubated overnight at 37°C. Next day plates were observed for bacterial growth and the lowest concentration showing complete killing of bacterium was termed as MBC of that complex.

## 2.3. Synthesis

### 2.3.1. Synthesis of $[\text{Ag}_2(1,5\text{-nds})(\beta\text{-Pic})_4]\cdot 2\text{H}_2\text{O}$ , **1**

$\text{AgNO}_3$  (0.5g, 3 mmol) was dissolved in 20 mL of distilled water. The beaker was covered with carbon paper as  $\text{AgNO}_3$  is light sensitive. A mixture of 1,5-naphthalenedisulfonic acid (0.43 g, 1.50 mmol) and sodium hydroxide (0.12 g, 3 mmol) in distilled water was stirred for 15 minutes in another beaker. On mixing both the solutions, white coloured precipitate appeared immediately. The precipitated product was filtered in a filtration unit under vacuum (in dark), washed with distilled water and dried. The isolated product,  $\text{Ag}_2(1,5\text{-nds})$ , was suspended in a mixture of methanol-water (4:1 v/v) and  $\beta\text{-pic}$  was added dropwise to the solution until clear colourless solution was obtained. When the solution was allowed to evaporate slowly at room temperature, white coloured crystals were obtained after one day. Crystals were filtered using fine filter paper and air dried in dark (yield=73%). Complex **1** was partially soluble in water but soluble in organic solvents. The complex **1** decomposed at 203°C. FT-IR ( $\text{cm}^{-1}$ ): 3414, 3055, 1605, 1480, 1451, 1384, 1338, 1241, 1182, 1125, 1051, 1029.  $^1\text{H}$  NMR (400 MHz, DMSO):  $\delta$ = 8.88(d, 2H, J= 8.8 Hz), 8.44(d, 8H), 7.95(d, 2H, J= 7.94), 7.73(d, 4H, J= 7.96), 7.43-7.38(m, 6H), 2.31(s, 12H).  $^{13}\text{C}$  NMR (400 MHz,  $\text{d}_6\text{-DMSO}$ ):  $\delta$ =150.54, 147.55, 143.69, 137.78, 133.86, 129.49, 129.06, 124.05, 123.98, 123.90, 40.08, 39.88, 39.67, 39.46, 39.25, 39.04, 38.83, 17.87. Anal. calcd. for  $(\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2)_2\cdot 2(\text{C}_{12}\text{H}_{14}\text{AgN}_2)\cdot 2(\text{H}_2\text{O})$ ; C, 44.80; H, 3.73; N, 6.15, Ag, 23.69%; Found; C, 44.48, H, 3.58; N, 6.42, Ag, 23.51%.

### 2.3.2. Synthesis of $[\text{Ag}_2(1,5\text{-nds})(\gamma\text{-Pic})_4]$ , **2**

Complex **2** was prepared in similar manner as complex **1** but using  $\gamma\text{-pic}$  instead of  $\beta\text{-pic}$ . When the solution was allowed to evaporate slowly at room temperature, white coloured crystals were obtained after two days. Crystals were filtered as described earlier and air dried in dark (yield=83%). Complex **2** was partially soluble in water but soluble in organic solvents. The melting point of the product is 203 °C. FT-IR ( $\text{cm}^{-1}$ ): 3414, 3055, 1605, 1480, 1451, 1384, 1338, 1241,

1182, 1125, 1051, 1029, 797, 702, 605, 524, 463. Anal. calcd. for  $(C_{10}H_6O_6S_2 \cdot 2(C_{12}H_{14}AgN_2) \cdot 6(H_2O))$  C, 41.52; H, 4.68; N, 5.69, Ag, 21.95%; Found; C, 41.38; H, 4.78; N, 5.42, Ag, 30.98%.

### 2.3.3. Synthesis of $[Ag_2(2,6-nds)(\beta-Pic)_4]$ , **3**

Complex **3** was prepared by similar synthetic procedure as adopted in the case of complex **1** but using disodium 2,6-naphthalenedisulfonate instead of solution of 1,5-naphthalenedisulfonic acid and sodium hydroxide (1:2) same N-donor ligand *i.e.*  $\beta$ -pic. When the solution was allowed to evaporate slowly at room temperature, white coloured crystals were obtained after one day. Crystals were filtered using fine filter paper and air dried in dark (yield=85%). Complex **3** was partially soluble in water but soluble in organic solvents. The complex **3** decomposed at 207 °C. FT-IR ( $cm^{-1}$ ): 3274, 1675, 1615, 1502, 1430, 1384, 1238, 1185, 1031, 790, 784, 605, 522, 485.  $^1H$  NMR (400 MHz, DMSO):  $\delta$ = 8.88(d, 2H, J= 8.72 Hz), 8.48(d, 8H, J= 5.48), 7.95(d, 2H), 7.73(d, 4H, J= 7.96), 7.43-7.35(m, 6H), 2.36(s, 12H).  $^{13}C$  NMR (400 MHz,  $d_6$ -DMSO):  $\delta$ =150.28, 149.00, 143.64, 129.48, 129.06, 125.45, 124.08, 123.92, 40.06, 39.85, 39.64, 39.43, 39.23, 39.02, 38.81, 20.57. Anal. calcd. for  $(C_{10}H_6O_6S_2 \cdot 2(C_{12}H_{14}AgN_2))$ ; C, 46.65; H, 3.88; N, 6.40, Ag, 24.66%. Found; C, 46.48; H, 3.92; N, 6.50, Ag, 24.98%.

### 2.3.4. Synthesis of $[Ag_2(2,6-nds)(\gamma-Pic)_4] \cdot 2H_2O$ , **4**

Complex **4** was prepared in similar manner as complex **3**, but employing 2,6-naphthalenedisulfonic acid. Crystals of complex **4** were isolated from the reaction mixture after three days which were separated and air dried in dark (yield=75%). Complex **4** was partially soluble in water but soluble in organic solvents. It decomposed at 210°C. FT-IR ( $cm^{-1}$ ): 3471, 3066, 1947, 1854, 1612, 1502, 1440, 1324, 1279, 1182, 1139, 1083, 1022, 805, 656, 537, 486. Anal. calcd. for  $(C_{10}H_6O_6S_2 \cdot 2(C_{12}H_{14}AgN_2) \cdot 2(H_2O))$  C, 44.80; H, 4.17; N, 6.15, Ag, 23.69%; Found; C, 40.70; H, 3.92; N, 6.42, Ag, 23.48%;.

## 2.4. X-crystallography

The crystallographic data for the **1-4** silver (I) complexes were collected on a Nonius Kappa CCD diffractometer at room temperature using graphite-monochromated Mo-K $\alpha$  radiation ( $\lambda$ = 0.71073



Å). Data sets were corrected for Lorentz-polarization and absorption effects [23]. The crystal parameters and other experimental details of the data collections are summarized in Table 1. The structures were solved by direct methods (SIR97) [24] and refined by full-matrix least-squares methods with all non-hydrogen atoms anisotropic. In complex **4**, O1 and O2 atoms were found to be disordered over two equivalent positions. Hydrogen atoms were included on calculated positions, riding on their carrier atoms, apart from those belonging to co-crystallized water molecules that were located in difference Fourier map and refined isotropically.

All calculations were performed using SHELXL-97 [25] implemented in the WINGX system of programs [26]. Selected bond distances and angles and geometrical parameters for hydrogen bonding interactions are given in Tables 2 and 3, respectively.

**Table 1. Crystallographic details and refinement parameters of complexes 1-4**

Crystal data	1	2	3	4
Chemical formula	$C_{10}H_6O_6S_2 \cdot 2(C_{12}H_{14}AgN_2) \cdot 2(H_2O)$	$C_{10}H_6O_6S_2 \cdot 2(C_{12}H_{14}AgN_2) \cdot 6(H_2O)$	$C_{10}H_6O_6S_2 \cdot 2(C_{12}H_{14}AgN_2)$	$C_{10}H_6O_6S_2 \cdot 2(C_{12}H_{14}AgN_2) \cdot 2(H_2O)$
$M_r$	910.54	982.61	874.51	910.54
Crystal system, space group	Triclinic, $P-1$	Triclinic, $P-1$	Monoclinic, $P2_1/n$	Triclinic, $P-1$
Temperature (K)	295	295	295	295
$a, b, c$ (Å)	8.7962 (2), 10.9643 (2), 10.9775 (2)	6.8310 (1), 11.0168 (2), 14.6514 (3)	7.6509 (2), 19.9220 (5), 11.4105 (3)	8.4944 (1), 10.0604 (1), 12.3587 (2)
$\alpha, \beta, \gamma$ (°)	66.2640 (11), 77.2230 (13), 69.1810 (14)	74.1850 (6), 78.6310 (7), 81.1920 (7)	94.7210 (13)	67.7770 (7), 83.7170 (7), 66.6530 (8)
$V$ (Å <sup>3</sup> )	902.10 (3)	1034.26 (3)	1733.30 (8)	896.62 (2)
$Z$	1	1	2	1
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	1.26	1.11	1.30	1.26
Crystal size (mm)	0.29 × 0.23 × 0.15	0.41 × 0.26 × 0.12	0.55 × 0.15 × 0.09	0.35 × 0.23 × 0.20
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	16585, 3790, 3507	22532, 4924, 4236	17894, 3927, 3394	15666, 3515, 3164
$R_{int}$	0.128	0.037	0.056	0.031
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.061, 0.146, 1.20	0.036, 0.108, 1.04	0.033, 0.102, 0.97	0.056, 0.161, 1.05
No. of reflections	3790	4924	3927	3515
No. of parameters	235	268	217	253
$D_{p_{max}}, D_{p_{min}}$ (e Å <sup>-3</sup> )	0.99, -0.65	0.50, -0.89	0.59, -0.77	2.65, -2.26

**Table 2. Selected bond distances and bond angles (Å, °) of complexes 1-4**

Complexes	1	2	3	4
<b>Bond distances</b>				
Ag1–N1	2.165(4)	2.132(2)	2.154(2)	2.135(5)
Ag1–N2	2.164(4)	2.131(2)	2.151(2)	2.138(4)
Ag1–O1	2.632(4)	2.705(2)	2.727(2)	2.708(9)
Ag1...O1W	2.929(5)			
S1–O1	1.456(4)	1.450(2)	1.453(2)	1.538(21)
S1–O2	1.459(3)	1.447(2)	1.438(2)	1.498(26)
S1–O3	1.448(5)	1.448(2)	1.440(2)	1.433(5)
S1–C13	1.780(3)	1.788(2)	1.786(2)	1.779(4)
<b>Bond angles</b>				
N1–Ag1 – N2	173.7(1)	168.86(8)	170.81(8)	165.8(2)
N1–Ag1 – O1	89.7(1)	89.57(7)	90.10(7)	99.3(7)
N2–Ag1 – O1	90.9(1)	101.55(6)	92.39(7)	94.7(7)

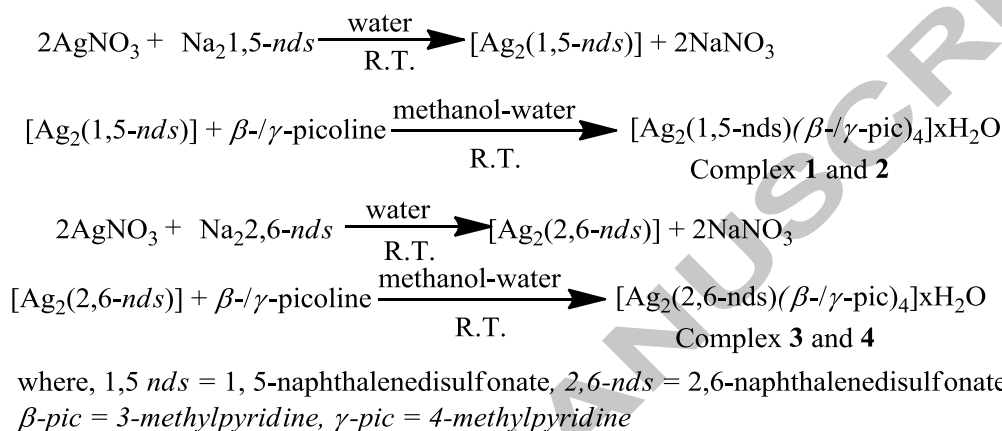
**Table 3. Hydrogen-bonding parameters (Å, °) of complexes 1-4**

D–H...A	D–H (Å)	H...A (Å)	D...A(Å)	∠DH...A (°)
<b>Complex 1</b>				
C1–H...O1W	0.93	3.254(5)	2.44	144
C5–H...O1	0.93	3.248(8)	2.53	133
C7–H...O1W	0.93	3.378(8)	2.59	142
C11–H...O1	0.93	3.262(5)	2.55	133
O1W–H...O3 <sup>i</sup>	0.98(6)	3.057(5)	2.18(5)	146(5)
O1W–H...O1 <sup>iii</sup>	0.82	2.870(3)	2.11	155
Equivalent positions: (i) -x,1-y,-z; (ii) x+1,y,z				
<b>Complex 2</b>				
C1–H...O1	0.93	3.233(3)	2.50	135
C7–H...O2	0.93	3.262(3)	2.33	177
O1W–H...O2	0.87(2)	2.750(3)	1.88(2)	176(2)
O2W–H...O3	0.85(3)	2.849(3)	2.01(3)	173(2)
O2W–H...O3W	0.86(5)	2.811(3)	2.04(5)	147(5)
O3W–H...O1W	0.87(6)	2.894(4)	2.02(6)	170(5)
O3W–H...O1W <sup>i</sup>	0.87(3)	2.873(4)	2.01(2)	173(3)
C8–H...O3W <sup>i</sup>	0.93	3.562(4)	2.63	174
O1W–H...O2W <sup>ii</sup>	0.87(3)	2.809(3)	1.94(3)	172(3)
C1–H...O1	0.93	3.233(3)	2.50	135
C7–H...O2	0.93	3.262(3)	2.33	177
Equivalent positions: (i) -x,1-y,2-z; (ii) x-1,y,z				
<b>Complex 3</b>				
C3–H...O2 <sup>i</sup>	0.93	3.364(4)	2.56	145
C11–H...O3 <sup>ii</sup>	0.93	3.472(3)	2.65	146
C15–H...O3 <sup>iii</sup>	0.93	3.390(3)	2.54	151
Equivalent positions: (i) x-1/2,-y-1/2,z-1/2; (ii) 1-x+1,-y,-z; (iii) x+1,y,z				
<b>Complex 4</b>				
O1W–H...O2	0.88	2.81(3)	1.97	156
C11–H...O1W <sup>i</sup>	0.93	3.49(1)	2.60	160
C6–H...O1W <sup>ii</sup>	0.96	3.542(8)	2.58	178
C17–H...O3 <sup>ii</sup>	0.93	3.495(7)	2.64	152
C12–H...O1W <sup>iii</sup>	0.96	3.41(1)	2.56	148
C15–HO3 <sup>iv</sup>	0.93	3.521(7)	2.67	150
Symmetry codes: (i) x,y+1,z; (ii) 1-x,-y,1-z; (iii) 2-x,-y,-z; (iv) x-1,y,z				

### 3. Results and Discussion

#### 3.1. Synthesis:

Complexes **1-4** were prepared by appropriate starting materials as described in experimental section and the summary of the diagrammatic representation of the synthesis is given below (Scheme 1).



#### Scheme 1. Schematic representation of syntheses of complexes 1-4

Elemental analyses of all the synthesized complexes **1-4** indicated the possible molecular formulae of newly synthesized silver (I) complexes. The complexes were further characterized by spectroscopic methods such as FT-IR, NMR *etc.* Finally, structures of complexes **1-4** were unambiguously determined by single crystal X-ray crystallography.

#### 3.2. Spectroscopic characterization:

##### 3.2.1. FT-IR spectroscopy

Infrared spectra of all the synthesized complexes **1-4** were recorded in the region 4000–400  $\text{cm}^{-1}$  and tentative assignments were made on the basis of earlier reports in literature [27,28]. In the complexes **1-4**, broad spectral peaks in the region 3600-3100  $\text{cm}^{-1}$  confirmed the presence of coordinated or lattice water molecules. The sharp peaks appeared in the region 3000-2850

$\text{cm}^{-1}$  in all four complexes were assigned to  $\text{sp}^2$  C-H stretching vibration of naphthalene or picoline moieties. The stretching frequency of C=C of aromatic naphthalene and picoline moieties appeared in the region  $1660\text{-}1620\text{ cm}^{-1}$  for all complexes. The assignments of peaks for  $\text{-SO}_3$  groups in all complexes **1-4** were made on the basis of previously reported complexes [16, 17]. Generally, the non-coordinated anionic sulfonate showed two bands due to asymmetric and symmetric stretching vibration modes [17]. In all four complexes, very little change in the stretching frequencies of  $\text{-SO}_{3(\text{asym})}$  and  $\text{-SO}_{3(\text{sym})}$  were observed, indicating the non-coordinating behavior of sulfonate anion. The rest of absorption bands in the region from  $800\text{-}500\text{ cm}^{-1}$  may be assigned to bending modes of C-C, C-H, N-H vibrations of picoline  $\text{-CH}_3$  group. The sharp peaks observed in the region  $530\text{-}470\text{ cm}^{-1}$  were assigned to Ag-N or Ag-O peaks. The FT-IR spectra of complexes **1-4** are given in Figure. 1.

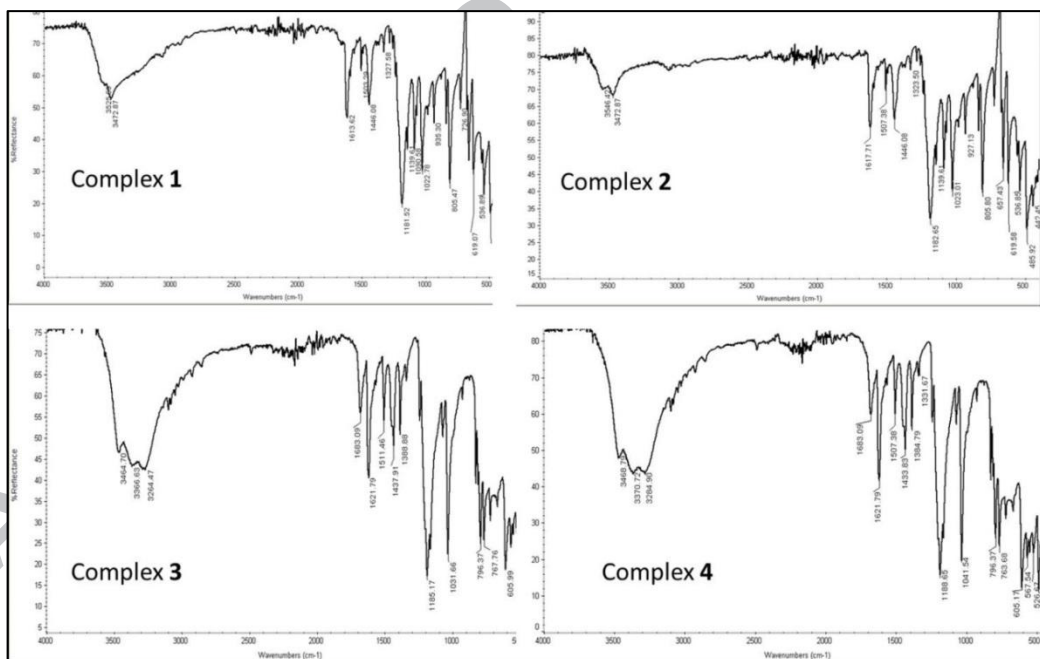


Figure 1. FT-IR spectra of complexes 1-4

### 3.2.2. Nuclear Magnetic Resonance spectroscopy

$^1\text{H}$  and  $^{13}\text{C}$ NMR spectra of the two representative complexes **1** and **3** were recorded in  $\text{d}^6$ -DMSO. The assignments for the various peaks in NMR spectra were done on the basis of reported literature [28, 29]. The spectra of complexes **1** and **3** showed characteristic  $^1\text{H}$  NMR peaks in the aromatic region corresponding to aromatic protons of 1,5-*nds* or 2,6-*nds* or  $\beta$ -/ $\gamma$ -picoline molecules in these complexes.

In the  $^{13}\text{C}$  NMR spectra, all aromatic carbons showed characteristic peaks in the range *i.e.*  $\delta$  120-150 ppm. The aliphatic carbons of methyl group of  $\beta$ -/ $\gamma$ -picoline (complexes **1** and **3**) showed peaks in the region 30-20 ppm in  $^{13}\text{C}$  NMR spectra. Spectral assignments were given in experimental section. The NMR spectra of complexes **1** and **3** were given as Fig. S1 and S2 (supplementary data).

### 3.3. X-ray crystal structure description

ORTEPIII [30] diagrams of complexes **1-4** are shown in Figure 2. In all these complexes, the asymmetric units are formed by one complex cation  $[\text{bis}(\text{picoline})\text{Ag}]^+$ , one half naphthalenedisulfonate anion and one (complexes **1** and **3**) or three (complex **2**) co-crystallized water molecules.

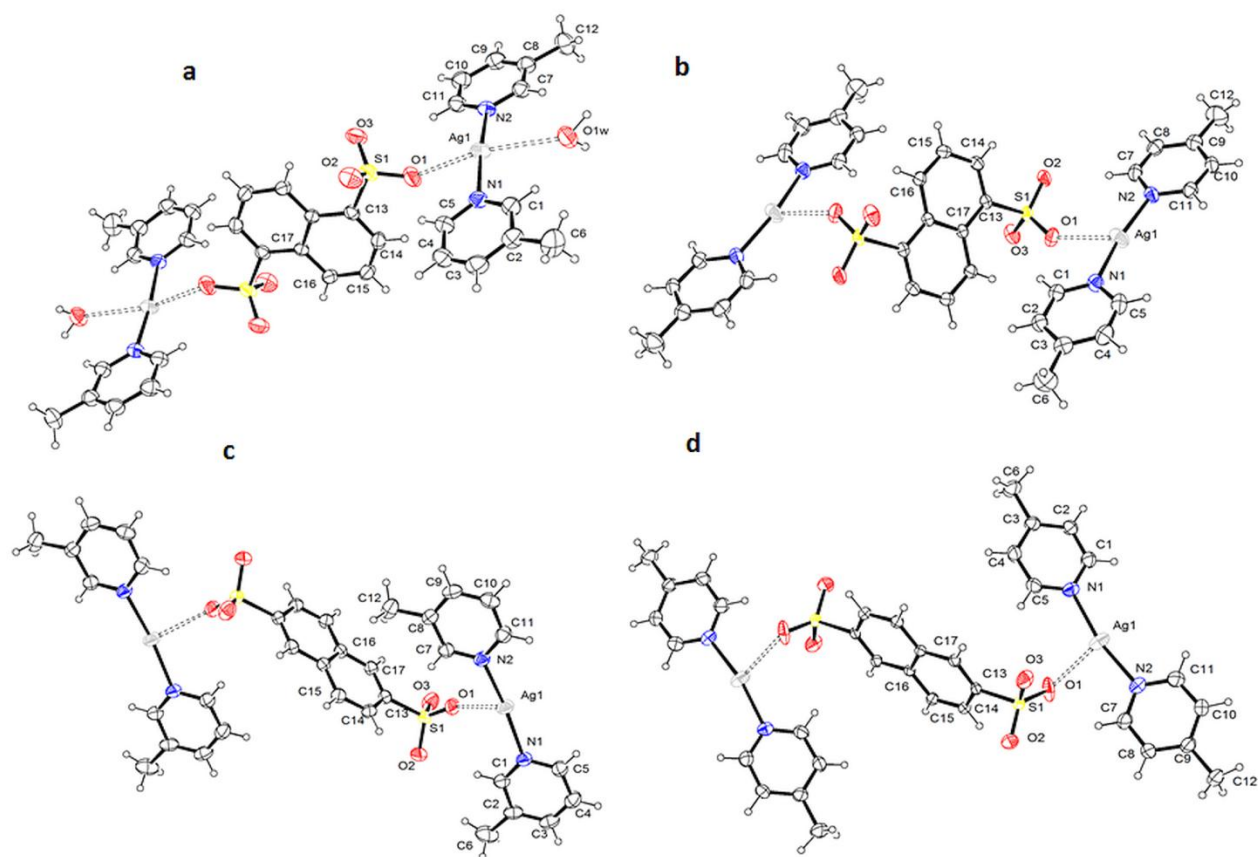
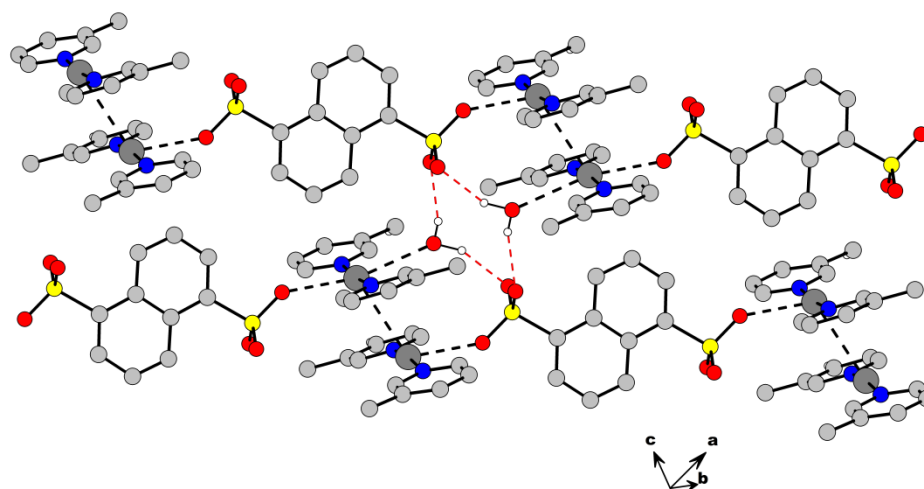


Figure 2. ORTEP diagram showing complexes **1-4** (a-d) with the atom numbering scheme and thermal ellipsoids at 40 % probability. Intermolecular short contacts are shown as dashed lines. In **2** and **4** (b, d) the water molecules are not shown for the sake of clarity.

The metal center is bound to the N atoms of two picoline molecules to give a linear geometry (Table 2), the N–Ag–N angle ranging in the narrow interval 173.7(1) - 165.8(2)°. 172.4(3)° is the mean value of the same angle calculated for all similar structures found in CSD (about 300 hits). Moreover, in all structures the Ag interacts with an oxygen atom of the sulfonate groups, with distances in the range 2.632(4)-2.727(2) Å (Table 2). These distances are not only longer than those found in Ag complexes with oxygenated ligands (mean 2.44(3) Å, calculated on 6254 hits), but also longer than the mean distance obtained for Ag–O(sulfonate) bond (2.50(3) Å, calculated on 968 hits); they could be therefore classified as short contacts. A similar Ag...O contact, involving the water molecule, has been found in complex **1**, so that the coordination sphere of the metal turns out to be expanded from 2 to 4 (Fig. 2a).

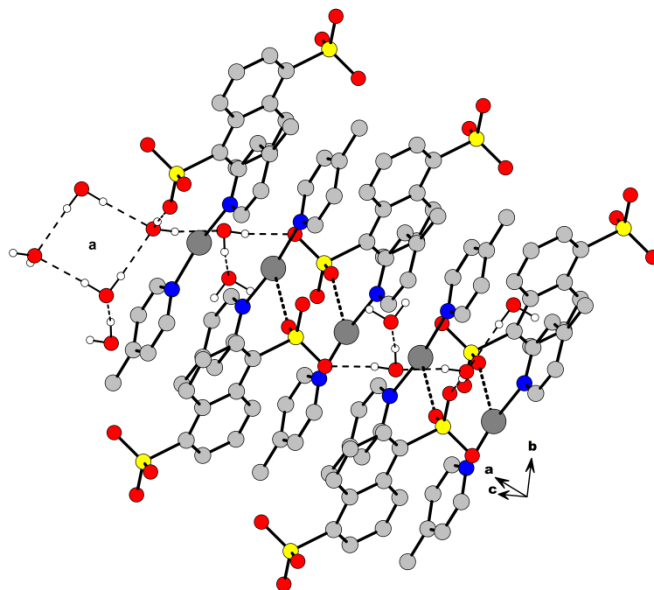
In spite of the similarity of the ligands, the direct comparison of the packing features is made complicated by the presence of different numbers of co-crystallized water molecules. Indeed, such solvent molecules act as a glue for the various constituents in the crystal lattice, being the only good hydrogen bonding donor groups. The packing diagram of complex **1** is shown in Figure 3. The disulfonate ions link two complexes on both ends of the molecule; in turn, two adjacent cations interact *via* argentophylic interactions, since the Ag1...Ag1(-x,1-y,-z) distance is 3.2295(3) Å, which is shorter than the sum of the van der Waals radii of Ag(I) ( $r_{\text{vdw}}(\text{Ag}) = 1.72 \text{ Å}$ ). Parallel infinite chains are so formed, and the occluded water molecules act as linkers between them. Besides some C–H...O weaker hydrogen bonding,  $\pi\cdots\pi$  interactions between the  $\beta$ -picoline rings of two interacting Ag complexes contribute to the crystal lattice stabilization: Cg(N1-C5)...Cg(N2-C11) = 3.610(2) Å, the ring normal and the vector between the ring centroids forming an angle of 16.7° [31]. Hydrogen bonding parameters are listed in Table 3.





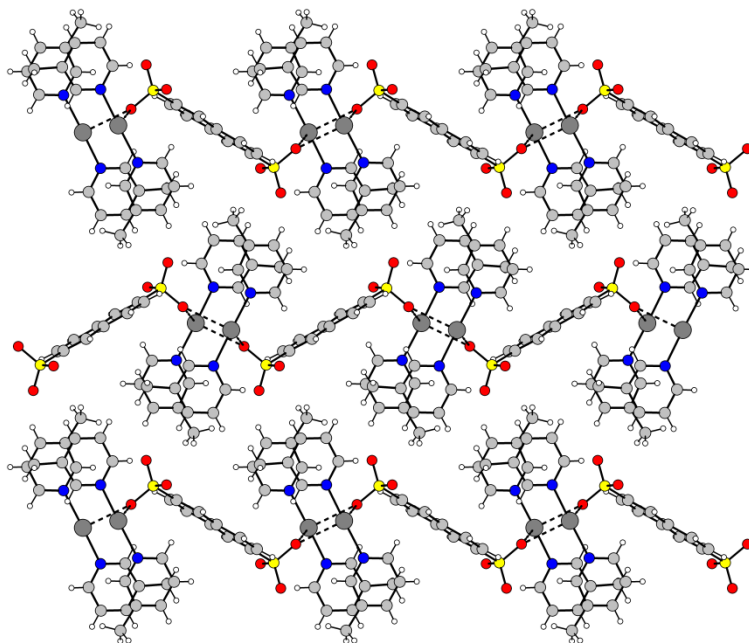
**Figure 3 Packing diagram of complex 1, showing one dimensional polymeric layers and two layers being interconnected by intermolecular hydrogen bonding through water molecule (in red). C-H hydrogen atoms have been omitted for the sake of clarity.**

The packing diagram of complex **2** is given in Figure 4. Again, the disulfonate anion is the linker between two cations, but in this case no metal...metal interaction is observed. The hydrogen bonding network is rather complicated, due to the number of co-crystallized water molecules, each acting both as donor and acceptor. Four of them are interacting with each other to form a characteristic R2,4(8) ring (**a** in Fig. 4).



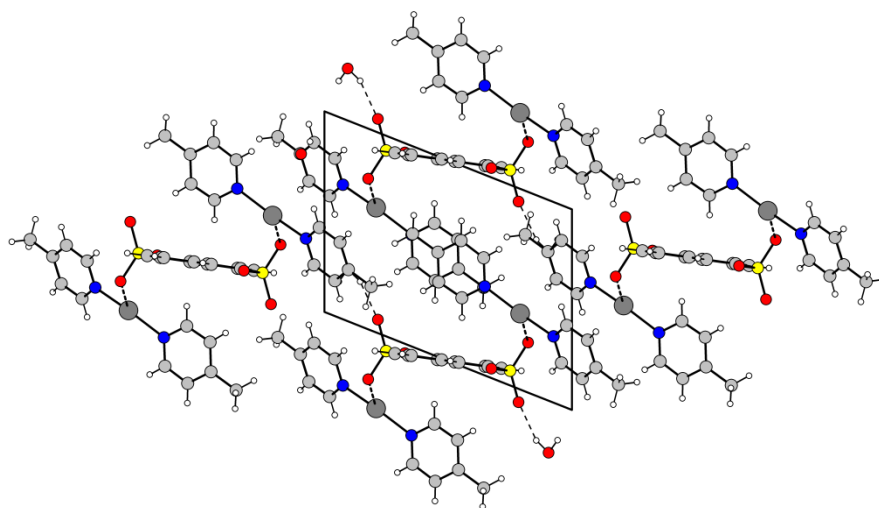
**Figure 4** Packing diagram of complex **2**. **a**= R<sub>2</sub>,4(8) ring formed by water molecules. C-H hydrogen atoms have been omitted for the sake of clarity. Hydrogen bonds are shown as dashed lines

The main characteristic of the packing architecture of **3** is the formation of one-dimensional layers in which both sulfonate groups of 2,6-nds act as coordinating sites to silver metal ion. One oxygen atom of sulfonate group is connected to two metal centers. The packing diagram of complex **3** is shown in Figure 5. No classical hydrogen bonds are present, due to the lack of good H-bond donors. Instead, the metal atoms are weakly interacting, with Ag...Ag(1-x,-y,-z) distance of 3.3893(3) Å and  $\pi\cdots\pi$  interactions between the picoline rings are established, with Cg1...Cg2(1-x,-y,-z) distance = 3.655(2) Å and the angle formed by the ring normal and the vector between the ring centroids = 11.7° (Cg1= centroid of the N1-C5 ring; Cg2= centroid of the N2-C11 ring).



**Figure 5 Packing diagram of complex 3 showing one dimensional polymeric layers (view along *a*). Hydrogen bonds are shown as dashed lines**

Complex **4** showed different and more simple packing features as compared to complexes **1-3**. The water molecules are involved in only one hydrogen bond with O2 atom, and the anionic 2,6-*nds* moieties are sandwiched between the cationic moieties. No relevant argentophylic or  $\pi\cdots\pi$  interactions are present. The packing diagram of complex **4**, viewed down *a* axis is shown in Figure 6.

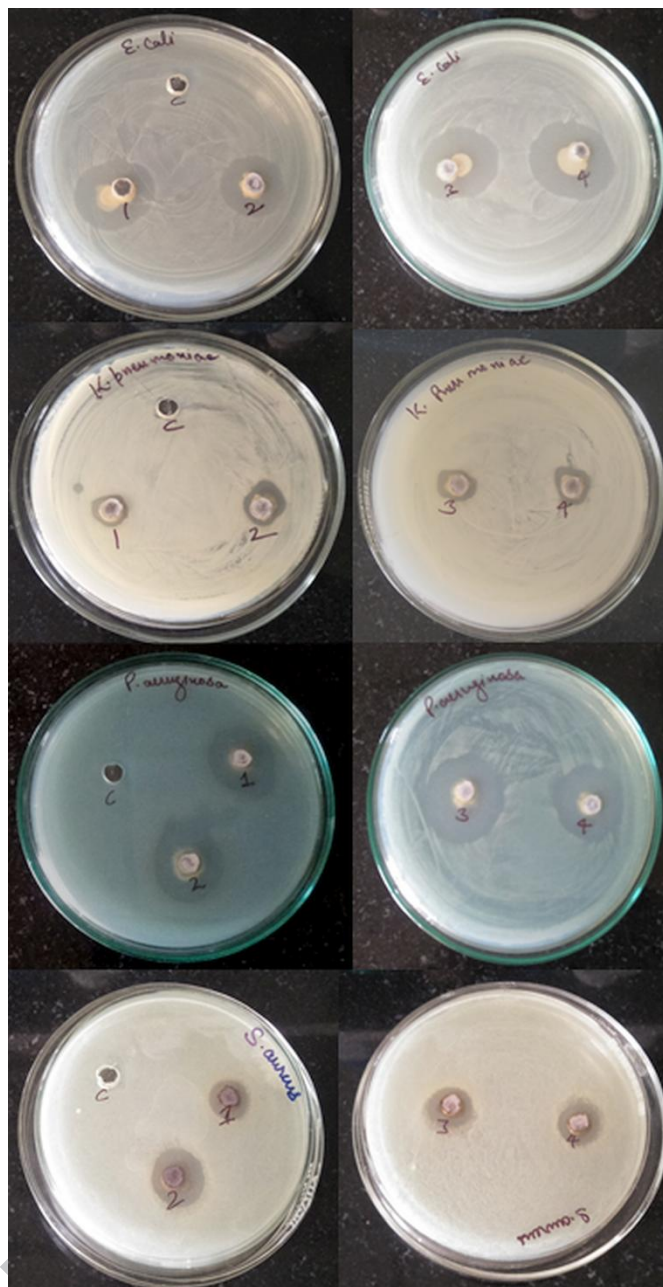


**Figure 6 Packing diagram of complex 4 showing stacking arrangement of cationic and anionic moieties. Hydrogen bonds are shown as dashed lines**

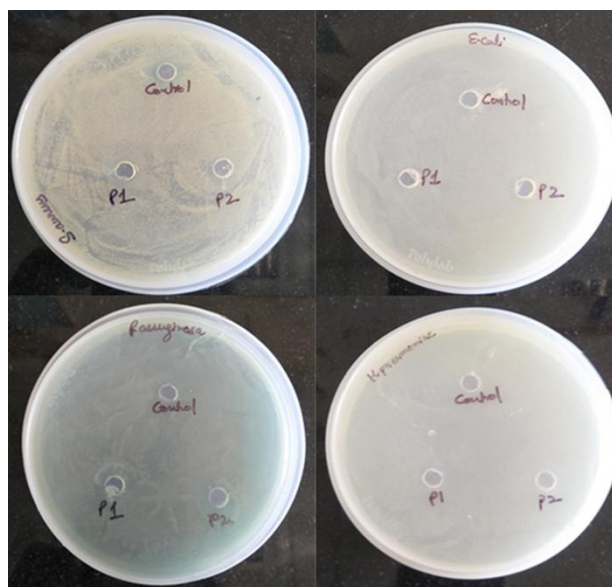
#### **4. Antibacterial activities**

##### **4.1 Agar well diffusion assay**

Agar well diffusion assay revealed potent antibacterial activity of all the four tested silver complexes **1- 4**. Solvent control, i.e. distilled water, did not exhibit any antibacterial activity whereas all the complexes showed significant antibacterial activity based on the zone of inhibition. Antimicrobial activity of all the complexes was relatively lower for *K. pneumoniae* and *S. aureus* than for *P. aeruginosa* and *E. coli*, as shown in Figure 7a. Antimicrobial activity of excipients, namely Na<sub>2</sub>[1,5-nds] (P1) and Na<sub>2</sub>[2,6-nds] (P2), was also evaluated; none of these salts was observed to exhibit antimicrobial activity in well diffusion assay against *K. pneumoniae*, *E. coli*, *S. aureus* and *P. aeruginosa* (Figure 8).



**Figure 7.** Images showing antibacterial activity of silver complexes against a range of bacterial pathogens.



**Figure 8. Images showing antibacterial potential of P1 and P2 compounds against a range of tested bacterium.**

#### **4.2 Minimum Inhibitory Concentration (MIC) and Minimum Bactericidal Concentration (MBC) determination**

The MIC and MBC values of each tested silver complexes and their starting material against a range of bacterial strains are presented in Tables 4 and 5. The disodium-*nds* salts showed sensitivity only at high concentration; this is consistent with their lower antibacterial efficacy compared to synthesized silver complexes, which, conversely, were highly effective against *E. coli* and *P. aeruginosa*. The highest MIC and MBC values for all the silver complexes are connected with the interaction with the other gram negative organism, *K. pneumoniae*, while variable MIC and MBC values were found for *S. aureus*. In this last case the organism was highly susceptible to complexes **1** and **2**, and less to complexes **3** and **4**.

**Table 4. MIC of tested silver complexes and P1, P2 salts against pathogenic bacterial strains.**

Complex Number	<i>K. pneumoniae</i> (mg/ml)	<i>S. aureus</i> (mg/ml)	<i>E. coli</i> (mg/ml)	<i>P. aeruginosa</i> (mg/ml)
<b>1</b>	2.5	0.62	0.31	0.12
<b>2</b>	1.25	0.62	0.31	0.31
<b>3</b>	1.25	1.25	0.12	0.12
<b>4</b>	1.25	1.25	0.12	0.31
<b>P1</b>	40	20	20	20
<b>P2</b>	40	20	40	40

**Table 5. MBC of tested silver complexes and P1, P2 salts against pathogenic bacterial strains.**

Complex Number	<i>K. pneumoniae</i> (mg/ml)	<i>S. aureus</i> (mg/ml)	<i>E. coli</i> (mg/ml)	<i>P. aeruginosa</i> (mg/ml)
<b>1</b>	2.5	1.2	0.62	0.62
<b>2</b>	2.5	1.2	0.62	0.31
<b>3</b>	2.5	1.2	0.31	0.31
<b>4</b>	2.5	2.5	0.31	0.62
<b>P1</b>	80	40	40	40
<b>P2</b>	40	40	40	80

## Conclusions

Synthesis and characterization of four new silver(I) naphthalenedisulphonate complexes  $[\text{Ag}_2(1,5\text{-nds})(\beta\text{-Pic})_4]\cdot 2\text{H}_2\text{O}$  (**1**),  $[\text{Ag}_2(1,5\text{-nds})(\gamma\text{-Pic})_4]\cdot 6\text{H}_2\text{O}$  (**2**),  $[\text{Ag}_2(2,6\text{-nds})(\beta\text{-Pic})_4]$  (**3**),  $[\text{Ag}_2(2,6\text{-nds})(\gamma\text{-Pic})_4]\cdot 2\text{H}_2\text{O}$  (**4**) with heterocyclic N-donor ( $\beta$ - and  $\gamma$ -picoline) ligands have been carried out. The structure of all complexes were determined by single crystal X-ray crystallography. The packing analyses clearly revealed the role of non-covalent interactions

(hydrogen bonds, including C-H...O bonds, argentophylic interactions,  $\pi\cdots\pi$  interactions) in crystal lattice stabilization. The antimicrobial activities of these complexes showed their high antibacterial efficacy against both gram positive and gram negative bacterial strains. Looking at the data reported in Tables 4 and 5, it seems difficult to find a correlation between the complex structures and the activity; results involving *E. coli* appear however interesting, since MIC and MCB for **1** and **2** are *ca.* twice the values found for **3** and **4**. This behaviour cannot be ascribed to the different chemical composition, so we would venture to guess that the molecules constituting the crystal in some way could retain in solution some of the interactions formed in the solid state and the resulting molecular aggregations, although transient, can interact differently with the bacteria.

#### Appendix A. Supplementary data

CCDC 1843292-1843295 contains the supplementary crystallographic data for **4**, **3**, **2** and **1**, respectively. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk).

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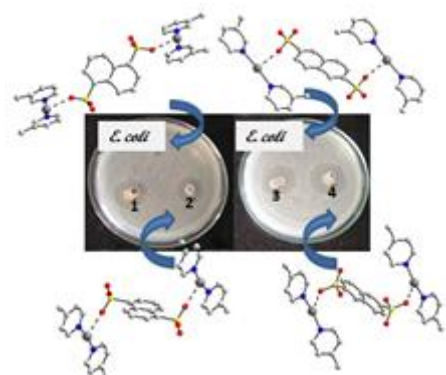


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Graphical Abstract

### Synopsis

Four new silver(I) naphthalenedisulfonate complexes with heterocyclic N-donor ligands have been synthesized and characterized through spectroscopic techniques and X-ray crystallography. Biological studies indicate they have antibacterial efficacy against both gram positive and gram negative bacterial strains.