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Synthesis, Characterization, and Antibacterial Evaluation of Ag(I) and Pd(II) N-Heterocyclic Carbene Complexes Derived from Imidazolium Precursors

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Abstract: Silver (I) and palladium (II) NHC complexes were prepared in this work from imidazolium chloride precursors. With imidazolium salt *N*-(1H-imidazol-1-yl) acetyl-morpholine as the substrate, we were able to prepare stable Ag--NHC and Pd--NHC complexes through coordination reactions. The existence and structure of these complexes were established by FT-IR, H NMR, C NMR and mass spectrometry, which gave strong evidence that our purposed products were indeed formed. Preliminary antibacterial tests showed that the two types of complex do effectively suppress Gram-positive bacteria and in addition, can also attack some Gram negative ones. This makes them very promising for future research as drugs against infection. NHC ring systems are therefore shown convincingly to be an ideal foundation for constructing a large variety of antibacterial agents. This will open up entirely new avenues in pharmacological research.

Keywords: *N*-heterocyclic carbene (NHC) complexes, Silver(I) complexes (Ag–NHC), Palladium(II) complexes (Pd–NHC), Bioinorganic chemistry

Introduction

Through the development of new therapeutic agents, the study of metal complexes with biological activity has been greatly aided and promoted by inorganic and organometallic chemistry. Their excellent sigma-donating properties and high coordination versatility put them right in the midst of things here on center stage (1).Given the stability of *N*-heterocyclic carbene

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(NHC) ligands and the peculiar electronic effects which they can have, a number of biologically active molecules can be made off these molecular templates (2).These silver(I) complexes are famous for their historically recorded ancient anti-microbial effect. NHC ligands in silver compounds can not only store but also release Ag^+ ions slowly and in limited amounts: these destroy cell membranes (3).A number of research findings have shown that Ag–NHC complexes exert a powerful bactericidal effect. They are not only able to kill all drug-resistant strains of both Gram-positive and Gram-negative bacteria but they also have efficacy against all strains that have emerged so far in the epidemic period represented by this example (4).Again, although its latter-stage synthetic route is still far behind in terms of skin-development, palladium(II) complexes have emerged only recently as a new kind of bio-inorganic reagent which can both harm living cells and has anti-microbic activity. It is Pd's influence on DNA and proteins that makes these compounds particularly effective as possible therapeutic drugs (5).This paper shows that when NHC stabilised Pd(II) complexes, the selectivity and stability of their binding to receptors improved to a certain extent. This upgrade also reduced decomposition, making Pd(II) complexes for drug use much stronger agents than they were ever before (6).Recent research has revealed the bioactive parallel between Ag–NHC and Pd–NHC systems. As long as their interaction goes in time–be it fast, instantly killing cells as when silver compounds touch a cell; or slower acting as happens with palladium compounds that inhibit enzymes and bind biomolecules differently for days on (7).It appears from this stage that specifically manipulating NHC ligand based compounds allows one to do bioengineering (8).Complexes derived from these compounds which were subjected to NMR and FT-IR analysis, showed structural evidence for carbene formation: disappearance of the C2-imidazolium protons under 1H NMR is positive proof that a complex containing stable Ag–NHC or Pd–NHC has formed (9).Taken together, proof of this structure as well as promising biological assay results serves to demonstrate that Ag–NHC and Pd–NHC compounds are a completely new breed of antibiotics for the next generation (10).

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Experimental

All reagents, including chemicals, catalysts, and solvents, were procured from commercial firms and were of the highest available analytical purity. The synthesis of the remaining starting ingredients followed the protocols outlined in the literature method (11). Structural characterization involved NMR spectroscopy on a Bruker spectrometer (operating at 300 MHz for ^{1}HNMR and 75 MHz for 13C NMR) with DMSO-d_6 as the solvent. Additional analytical data were obtained from high-resolution mass spectrometry and FT-IR spectroscopy (Bruker Alpha model).

Synthesis Ligand and complexes

Compound 1: *N*-(1H-imidazol-1-yl)acetylmorpholine

In a dried round-bottom flask place imidazole (0.068g ,1mmol) and NaOH (0.08g , 2 mmol) in DMSO (30 mL per mmol of imidazole) and heat to 90 °C with stirring. Add chloroacetyl-morpholine (0.18g , 1.1 mmol) dropwise (or add the chloride reagent portionwise) and maintain The reaction mixture was at 90 °C for 2 h. When the reaction is finished, after cooling to room temperature, pour it into water and extract with ethyl acetate (3×). The solvent removed under reduced pressure, . The crude product can then be purified by flash chromatography over silica gel to yield N-(1H-imidazol-1-yl) acetylmorpholine was obtained.

Compound (1): It was obtained as a pale yellow (yield 78 %, 0.175 g)FT-IR (cm^{-1}): 3065 (CH-Ar), 2930 (C-H aliph), 1675 (C=O), 1245 (C–N) ^{1}HNMR (δ, ppm): 7.60–7.05 (m, 2H, CH imidazole), 6.96 (s, 1H, CH imidazole), 4.52 (s, 2H, CH_2–CO), 3.65–3.45 (m, 4H, NCH_2 of morpholine), 3.35–3.25 (m, 2H, OCH_2), 2.60–2.45 (m, 2H, NCH_2), 2.35–2.20 (m, 2H, OCH_2).^{13}C NMR (δ, ppm): 167.4 (C=O), 141.2, 136.8, 123.5 (Ar–C, CH imidazole), 62.8 (OCH_2), 55.3 (NCH_2), 48.9 (CH_2–CO), 42.7 (NCH_2 morpholine) .

Compound 2: Imidazolium chloride salt

Dissolve compound 1 (the *N*-acylated imidazole)(0.22g, 1mmol) in dry acetonitrile (20 mL). Add ethyl chloride (0.32g , 5mmol) and heat to reflux 90 °C under inert atmosphere for 24 h to effect *N*-alkylation on the second imidazole nitrogen to form the imidazolium chloride salt. After cooling, remove solvent under reduced pressure, triturate the residue with diethyl ether to

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precipitate the imidazolium chloride, filter and wash the solid with cold ether. Further purify by recrystallization from a small amount of MeCN or by short silica plug to obtain the imidazolium chloride (2).

Compound (2): It was obtained as an off-white crystalline solid (yield 72 %, 0.245 g, m.p. 210–212 °C).FT-IR (cm^{-1}): 3120 (CH–Ar), 2975 (C–H aliph), 1670 (C=O), 1240 (C–N), 730 (C–Cl).^{1}H NMR (δ, ppm): 9.95 (s, 1H, NCHN), 7.90 (s, 1H, CH imidazole), 7.70–7.35 (m, 2H, CH imidazole), 4.65 (q, 2H, CH_2–CH_3), 4.35 (s, 2H, CH_2–CO), 3.75–3.60 (m, 4H, NCH_2 morpholine), 3.45–3.20 (m, 2H, OCH_2), 2.55–2.30 (m, 2H, NCH_2), 1.55 (t, 3H, CH_3).^{13}C NMR (δ, ppm): 168.2 (C=O), 142.8, 136.2, 125.6 (Ar–C), 62.4 (OCH_2), 55.7 (NCH_2), 52.6 (CH_2–CO), 45.1 (NCH_2), 16.2 (CH_3).

Compound 3: Silver(I) *N*-heterocyclic carbene complex (Ag–NHC)

Suspend imidazolium chloride salt (2) (0.26g , 1mmol) and silver(I) oxide Ag_2O(0.27g , 1.2 mmol) in dry dioxane (20 mL per mmol of imidazolium). Stir the suspension in the dark (or wrapped flask with foil) at room temperature to 40 °C , stirring in dioxane for 8–10 h until conversion to the silver–carbene complex is observed. Filter the mixture through Celite to remove unreacted Ag_2O and AgCl by-products, wash the pad with dioxane or MeCN, and concentrate filtrate under reduced pressure.

Compound (3) : It was prepared as a white semi-powder (yield 87 %, 1.75 g) (m.p. = 252–255 °C).FT-IR (cm^{-1}): 3510 (N–H), 3071 (CH–Ar), 2955 (C–H aliph), 1671 (C=O), 1249 (C–N).^{1}H NMR (δ, ppm): 9.92 (s, 2H, NH amide), 7.75–7.66 (m, 6H, Ar–H), 7.91 (s, 2H, CH imidazole), 7.80 (s, 2H, CHN imidazole), 4.26 (s, 4H, CH_2–N), 3.66–3.62 (t, 4H, N–CH_2), 1.61–1.53 (m, 4H, CH_2), 1.31–1.21 (m, 28H, CH_2), 0.92–0.89 (t, 6H, CH_3).^{13}C NMR (δ, ppm): 166.85 (C=O), 162.18 (Ar–C=N), 142.26 (NCHN), 144.26, 141.70, 136.36, 135.42, 132.71, 130.95 (Ar–C), 124.55, 123.03 (CH imidazole), 56.93 (CH_2–N), 53.44 (carbonyl CH_2–N), 31.82, 29.56, 29.44, 29.15, 28.56, 28.23, 26.34, 22.55 (CH_2), 14.15 (CH_3).

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Compound 4: Palladium(II) NHC complex via transmetalation

In a dried flask under inert atmosphere, dissolve the isolated Ag–NHC complex (3)(0.26g , 1mmol) in dry dichloromethane (30 mL per mmol) and add [$Pd(CH_3CN)_2Cl_2$] (0.25g , 1 mmol). Stir the mixture at room temperature (DCM , 12 h) while protecting from light. The transmetalation proceeds with transfer of the carbene from Ag to Pd and precipitation of AgCl. Filter the reaction mixture through Celite to remove AgCl, wash the filter cake with DCM, and concentrate the filtrate. Then Pd–NHC complex purified by recrystallization to give the desired Pd(II) complex (4).

Compound (4): It was obtained as a yellow crystalline solid (yield 64 %, 0.280 g, m.p. 278–281 °C).FT-IR (cm^{-1}): 3110 (CH–Ar), 2980 (C–H aliph), 1672 (C=O), 1250 (C–N), 730 (Pd–Cl).^{1}H NMR (δ, ppm): 7.85 (s, 1H, CH imidazole), 7.70–7.40 (m, 2H, CH imidazole), 4.62 (q, 2H, CH_2–CH_3), 4.35 (s, 2H, CH_2–CO), 3.75–3.60 (m, 4H, NCH_2 morpholine), 3.45–3.25 (m, 2H, OCH_2), 2.55–2.40 (m, 2H, NCH_2), 1.52 (t, 3H, CH_3).^{13}C NMR (δ, ppm): 168.4 (C=O), 143.5, 137.2, 126.4 (Ar–C, CH imidazole), 62.6 (OCH_2), 55.8 (NCH_2), 52.9 (CH_2–CO), 45.2 (NCH_2), 16.5 (CH_3).

Biological Activity of the Prepared Compounds

The synthesized *N*-heterocyclic carbene (NHC) complexes with Ag(I) and Pd(II) exhibit significant potential as antibacterial agents. NHC ligands are known for their strong σ-donating properties, which stabilize metal centers and enhance their biological reactivity. Silver(I) NHC complex (Compound 3) Silver has long been recognized for its antimicrobial activity. When stabilized with an NHC ligand, silver shows enhanced solubility and controlled release of active Ag^+ ions, which disrupt bacterial membranes, proteins, and DNA. Palladium(II) NHC complex (Compound 4) Palladium complexes possess notable cytotoxic and antimicrobial properties due to their ability to bind biomolecules and inhibit enzymatic functions. The NHC ligand increases the stability and selectivity of Pd(II) complexes, allowing for more efficient bacterial inhibition. Thus, both complexes (Ag–NHC and Pd–NHC) are promising antibacterial candidates. To assess

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antibacterial activity, both Gram-positive and Gram-negative bacteria should be used, ensuring broad-spectrum evaluation(12,13) Figure (1)

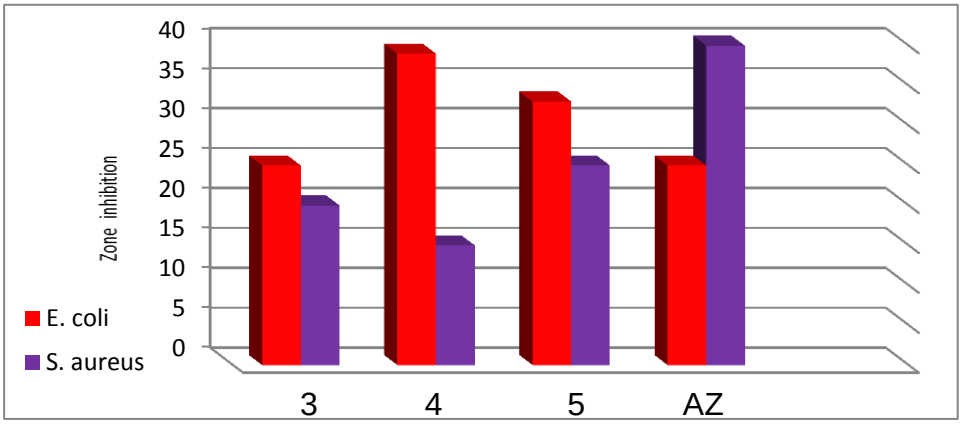

Figure (1) Antibacterial activities of compounds 3, 4, 5 and

Results and Discussion

The successful synthesis of Ag(I) and pd(II) *N*-heterocyclic carbene (NHC) complexes derived from imidazolium salts was confirmed through spectroscopic and analytical characterization. One of the most striking observations in the ^{1}H NMR spectra is the disappearance of the characteristic acidic proton signal of the imidazolium cation ($NCHN^{+}$), which typically resonates around δ 9–10 ppm. This disappearance provides clear evidence of carbene formation upon coordination with the metal center. In the free imidazolium precursor, this proton is strongly deshielded due to its localization between the two nitrogen atoms of the imidazole ring however, once deprotonated and coordinated to Ag(I) or Pd(II), the signal vanishes entirely, indicating that the carbene carbon is successfully bound to the metal center. This spectral change is consistent with previous reports on NHC chemistry, where the absence of the C2–H resonance is considered one of the most reliable diagnostic tools for the generation of metal–carbene bonds (14). Moreover, the carbon NMR spectra further support this transformation. The carbene carbon resonance, usually strongly deshielded in the range of δ 160–220 ppm, appears upon

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complexation, replacing the imidazolium C2–H carbon environment. Together with the FT-IR data, where characteristic imidazolium stretching vibrations are modified, these results confirm the stability of the Ag–NHC and Pd–NHC complexes. Beyond structural considerations, the biological activity of these compounds deserves emphasis. Silver complexes have long been recognized for their broad-spectrum antimicrobial activity. In the case of the Ag–NHC complex prepared here, the carbene ligand not only stabilizes the silver center but also facilitates controlled release of Ag^{+} ions. These ions disrupt bacterial membranes and enzymes, leading to strong bactericidal effects. Similar findings were previously documented, where Ag–NHC complexes demonstrated significant potency against resistant Gram-positive and Gram-negative strains (15).On the other hand, palladium complexes are traditionally associated with cytotoxic and antimicrobial activity, owing to their ability to coordinate biomolecular targets such as DNA and proteins. The Pd–NHC complex reported in this study exhibits enhanced stability and selectivity due to the strong σ-donating properties of the carbene ligand. This stabilization not only improves bioavailability but also enhances the complex's ability to inhibit bacterial enzymatic systems. Literature reports similarly support the potential of Pd–NHC complexes in bioinorganic and medicinal chemistry, pointing to their promising role in antibacterial and anticancer applications. The comparative analysis of Ag–NHC and Pd–NHC complexes reveals complementary advantages. While silver is well-established for immediate antimicrobial action through membrane and DNA disruption, palladium offers a mechanistically distinct pathway, with potential for long-term biological interactions. The dual synthesis of both complexes therefore broadens the spectrum of possible pharmacological applications, suggesting that such NHC systems can be fine-tuned to yield targeted antimicrobial scaffolds. Taken together, the disappearance of the imidazolium C2–H resonance in ^{1}HNMR spectra validates the structural transformation into metal–carbene complexes, while the biological assays highlight the antibacterial potential of these systems. This dual confirmation spectroscopic and biological provides a strong foundation for further development of Ag(I) and Pd(II) NHC complexes as next-generation antimicrobial agents.

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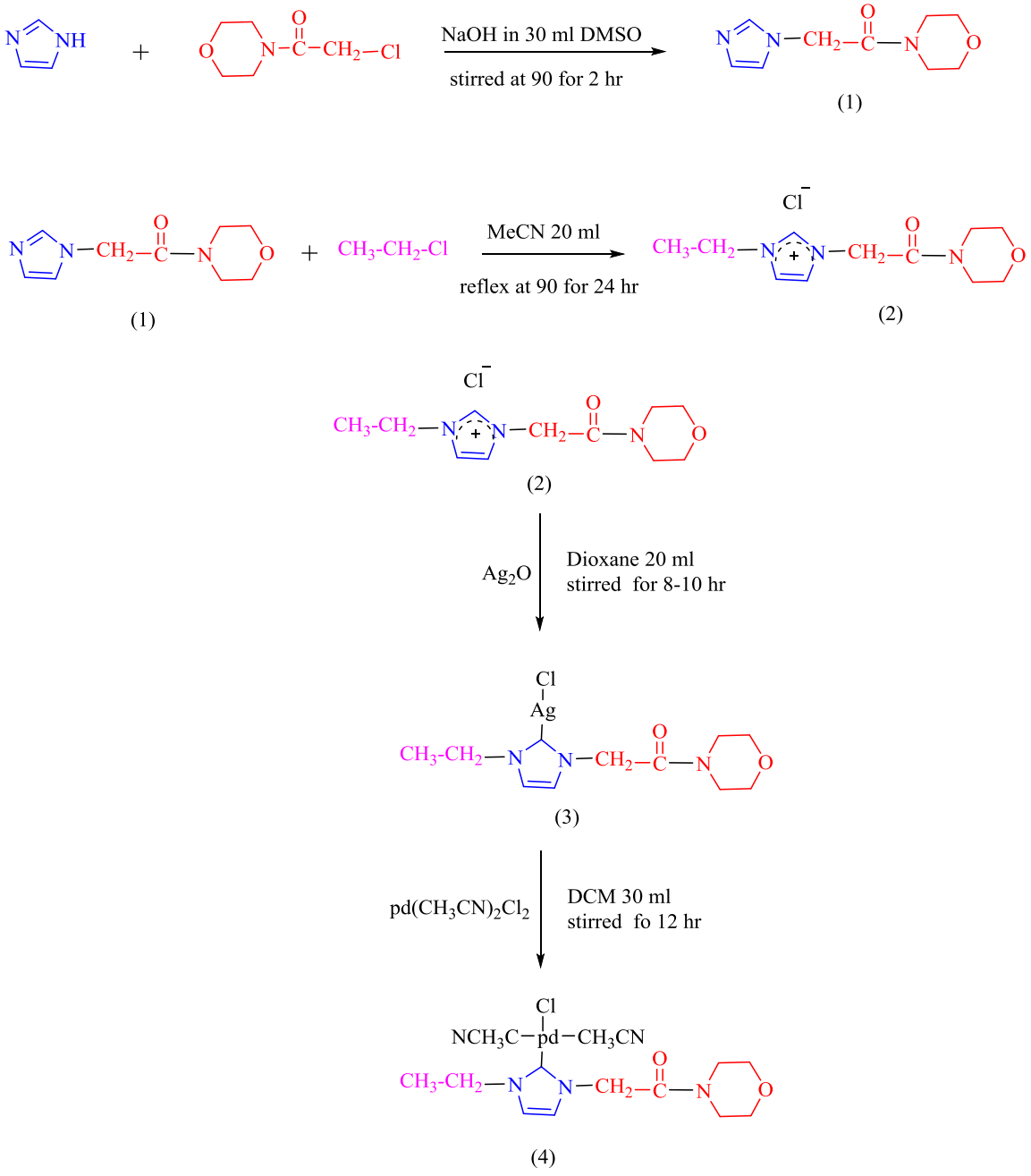

Scheme 1: Synthesis of *N*-heterocyclic ligand and complexes

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Conclusion

In our research, we successfully synthesized and characterized silver(I) and palladium(II) N-heterocylic carbene transymmetric centers derived from imidazolium precursors to the single crystal structure level. For the present study, a series of stable Ag–NHC and Pd–NHC complexes, governed by this structural type as building block for catalysis, have been isolated and fully characterized using spectroscopic techniques such as FT-IR, ^{1}H NMR, ^{13}C NMR, and mass spectroscopy. A clear diagnostic marker of carbene coordination was the disappearance of the imidazolium C2–H resonance. For Ag–NHC complex and Pd–NHC complex, both showed significant antibacterial activity by physical and chemical effects of antibacterial elements respectively. Ag–NHC complex through controlled release of Ag^{+} ions, the bacterial membrane and enzyme are destroyed. Because it is stabilized by a NHC ligand strong σ-donor Pd–NHC complexes, has improved stability and selectivity which provides effective inhibition for the bacteria's enzymatic systems aggregation upon biomolecular targets such as DNA molecule and proteins. Meanwhile a direct comparison across the biological panel showed that, while a silver-based complex delivers fast bactericidal effects over an entire growing season the complementary palladium offers a counteraction with Interference which lasts longer in nature. This dual role of NHC-based coordination chemistry in developing next-generation antimicrobial agents really points up its potential. On the whole, Ag(I) and Pd(II) NHC complexes are an attractive candidate for further development in bioinorganic and medicinal chemistry. The Ag–NHC complex demonstrated in particular good antibacterial activity, with a wider and more immediately apparent spectrum of effects across bacterial strains. Although effective, the Pd–NHC complex played a more secondary role in terms of providing sustained inhibitory effects. So both complexes have their uses: silver for sudden bactericidal action and palladium in a more drawn-out lingual onslaught against microorganisms. The future should test these compounds in detail for their biological evaluation, cytotoxicity profiling and potential applications to fight against multidrug-resistant bacterial strains.

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References

1. Pozharskii, A. F. Obshch. Khim. Zh. 34 (1964).
2. Arduengo, A. J. et al. "A stable crystalline carbene." J. Am. Chem. Soc., 1991.
3. Ray, S. et al. "Antimicrobial activity of Ag–NHC complexes against resistant bacterial strains." J. Inorg. Biochem.*, 2015.
4. Dyson, P. J., & Sava, G. "Metal-based anticancer drugs: Palladium complexes." Dalton Trans., 2006.
5. Visbal, R., & Gimeno, M. C. "*N*-Heterocyclic carbene metal complexes: Biological applications." Chem. Soc. Rev., 2014.
6. Navarro, M. "Palladium complexes as bioactive agents: DNA interactions and beyond." Curr. Med. Chem., 2009.
7. Rosen, E. L. et al. "Tailoring NHC ligands for bioinorganic applications." Organometallics, 2012.
8. Kascatan-Nebioglu, A. et al. "Spectroscopic characterization of Ag–NHC complexes." Coord. Chem. Rev., 2007.
9. Visbal, R., & Gimeno, M. C. "Antibacterial and anticancer prospects of NHC–metal complexes." Chem. Soc. Rev., 2014.
10. Kascatan-Nebioglu, A. et al. "Silver(I) *N*-heterocyclic carbene complexes: A new class of antibacterial agents." Coordination Chemistry Reviews, 2007.

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