
Preparation, Physical Characterization and Antibacterial Activity of Ni (II), Cu (II), Co (II), Cd (II), Zn (II) and Cr (III) Schiff Base Complex Compounds

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Abstract: A Schiff base (SB) derived from p-hydroxy benzaldehyde and 4-aminobenzoic acid. The transition metal complexes of Ni (II), Cu (II), Co (II), Cd (II), Zn (II) and Cr (III) were prepared separately with the Schiff base (SB), which were used as ligand. Several physical tools, in particular; elemental analysis, molar conductivity, magnetic susceptibility, infrared spectroscopy (IR), electronic absorption spectroscopy (ESR) to investigate the chemical structure of the prepared transition metal complexes. The elemental analysis data show the formation of 1:2 [$M^1:2L$] and 1:3 [$M^2:3L$] complexes of the formula of $M^{2+}L_2$ and $M^{3+}L_3$, respectively where $M^{2+} = Ni(II), Cu(II), Co(II), Cd(II), Zn(II)$ and $M^{3+} = Cr(III)$ and $L =$ Schiff base (SB). The molar conductance (conductivity) measurements were revealed that all the complexes are nonelectrolyte in nature. The infrared (IR) spectral studies indicated the binding sites of the Schiff base ligand with the transition metal ions. The magnetic susceptibility measurements and electronic spectral results supported the predicted coordination geometry of the complexes and magnetic properties (para or dia-magnetic nature) of the complex compounds. The free Schiff base and its complexes have been tested for their antimicrobial activities against several human pathogenic (two gram-positive and two gram-negative) bacteria. The obtained results showed that the complex compounds exhibit moderate to strong antimicrobial activity compared with kanamycin and ampicillin.

Keywords: Schiff Base, Metal Complex, Spectral Studies, Antimicrobial Activity

1. Introduction

Schiff bases were first discovered by a German chemist, Nobel Prize winner, Hugo Schiff. Schiff base is a condensation product of any primary amine with an active carbonyl group of an aldehyde or a ketone under optimum conditions and contain the azomethine group ($-CH=N-$ or $>C=N-$). The structure of Schiff base implies that it (also known as imine or azomethine) is a nitrogen analogue of an aldehyde or ketone in which the carbonyl group (CO) has

been substituted through an imine or azomethine group [1, 2, 3]. The Chemistry of Schiff-base complexes has attracted a great deal of attention ever since Pfeiffer carried out his pioneering research in the 1930's. In organic chemistry Schiff bases are most extensively utilized in pigments and dyes industries, catalysts, intermediate in organic synthesis, and as a polymer stabilizer [4-6]. Furthermore, Schiff bases and their metal complexes are also well known to have pronounced biological activity [12-13] and form an important class of compounds in medicine and pharmaceutical field. It

also exhibited some degree of antibacterial, antifungal, antitumor, anticancer and anti-inflammatory activity [14-22], the azomethine linkage are responsible for the biological activity of Schiff bases. Transition metal complexes of these ligands exhibit varying configurations, structural liability and sensitivity to molecular environments. The central metal ions in these complexes act as active sites for pharmacological agent. This feature is employed for modelling active sites in biological systems [23-26]. Mixed -ligand complexes of Cu (II) containing the Schiff base ligand derived from 2-hydroxybenzaldehyde with 2-amino phenol/3-amino phenol and bidentate auxiliary ligands were synthesized and characterized by Kudrat *et. al* [3]. The authors observed that the complexes with Schiff bases exhibited the significant anti-microbial activity. Para-hydroxy benzaldehyde and 4-aminobenzoic acid compounds are capable to form complexes with transition metal ions in the form of Schiff bases. The Schiff bases are important class of ligands in coordination chemistry and play an important role as extensive applications in different fields [6-8]. A large number of the metal complexes with different electronic structures have been synthesized using Schiff base ligands [9-12]. In recent years, metal complexes of Schiff bases have attracted considerable attention due to their remarkable antibacterial, antifungal and antitumor activities [13-17]. Previously, we had synthesized few transition metals complexes of such type and studied their antimicrobial activity [18]. It was observed that the complexes have potential antimicrobial activity. Keeping these facts in view the significance of metal in biology, we here in report the synthesis of the Schiff base by the condensation of p-hydroxy benzaldehyde and 4-aminobenzoic acid and complexation this Schiff base with several transition metals Ni (II), Cu (II), Co (II), Cd (II), Zn (II) and Cr (III) ions. Also, we characterized of the complexes on the basis of elemental analysis, conductivity and magnetic measurements and infrared spectral and electronic spectroscopy to investigate the structure of the complexes. In addition, the antimicrobial activities of these complexes were also reported here.

2. Experimental

2.1. Materials

All the chemicals used in this work were reagent chemically pure and reagents grade (BDH/Aldrich). Solvents were purified and dried according to standard procedures. Melting points of all metal complexes were measured by an electro thermal melting point apparatus model no. AZ6512. Elemental analysis for carbon, hydrogen and nitrogen were performed by Perkin 2400 Organic Elemental Analyzer II at Kayama University, Japan. The SHERWOOD SCIENTIFIC Magnetic Susceptibility Balance was used for measuring magnetic susceptibility of the metal complexes. Infrared spectra as KBr disc were recorded with a NICOLET 310, FTIR Spectrophotometer, Belgium, from 4000-400 cm^{-1} in the Department of Metallurgy and Material Science, Bangladesh University of Engineering & Technology (BUET), Dhaka, Bangladesh. UV-visible spectra were recorded with a SHIMADZU DOUBLE BEAM spectrophotometer (model UV-1200) from 200-900 nm in Central Science Laboratory, University of Rajshahi, Rajshahi-6205, Bangladesh.

2.2. Preparation of Schiff Base

The Schiff base was prepared by mixing stoichiometric ratio of p-hydroxy benzaldehyde in ethanol with ethanolic solution of 4-aminobenzoic acid. p-hydroxybenzaldehyde (1.37 g, 10 mmol) in absolute ethanol (20 mL) was added to an ethanolic (30 mL) solution of 4-aminobenzoic acid (1.22 g, 10 mmol). The mixture was stirred for 4 hours at ambient temperature. Then it was allowed to stand for 5 days at room temperature. The resulting solution was evaporated under vacuum to remove the solvent. The Product was collected by filtration, washed several times with ethanol and recrystallized from hot ethanol. The melting point of the product found to be 232°C, and its purity was confirmed by TLC technique [27]. A yellow precipitate was observed and then dried under vacuum desiccator over anhydrous CaCl_2 . The reaction scheme of the preparation of Schiff base are given below (Figure 1):

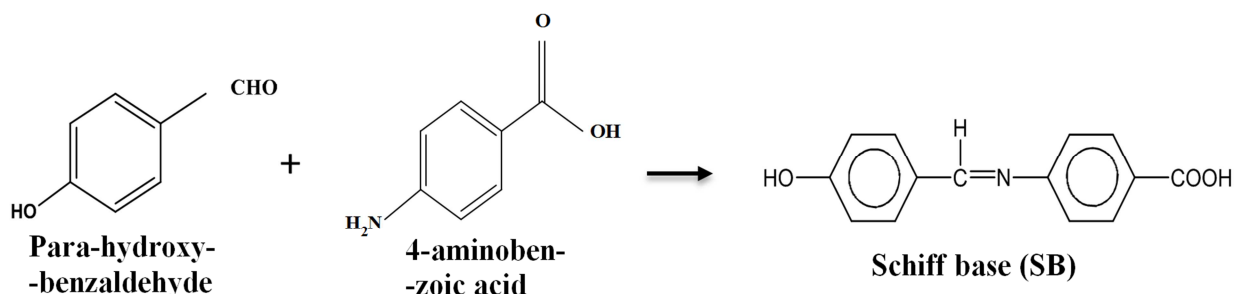


Figure 1. Reaction scheme for the preparation of Schiff base (SB).

The general mechanism of imine formation (scheme 2) begins as a nucleophilic addition to the carbonyl group. In this case, the nucleophile is the amine, which reacts with the aldehyde or ketone to give an instable addition product called

a carbinolamine. A carbinolamine is a compound with an amine group ($-\text{NH}_2$) $-\text{NHR}$ or $-\text{NR}$ and a hydroxyl group to the same carbon as in Figure 2.

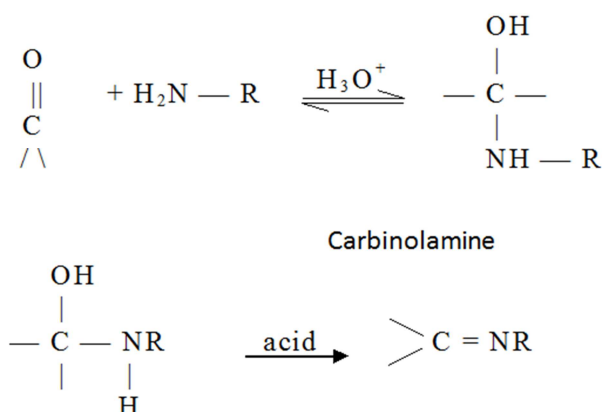


Figure 2. Reaction mechanism for the formation of azomethine group ($-\text{CH}=\text{N}-$ or $>\text{C}=\text{N}-$) in Schiff base.

2.3. Preparation of Schiff Base Complexes

1 mmol of salt Ni (NO₃)₂.6H₂O, Cu (NO₃)₂.3H₂O, CoCl₂.6H₂O, Cd (NO₃)₂.4H₂O, Zn (NO₃)₂.6H₂O and Cr (NO₃)₃.9H₂O were individually dissolved in 10 ml of absolute ethanol. The Schiff base (SB) solution was added to these salts solution separately and stirred for 4 hours at ambient temperature and allowed to stand for half an hour. The precipitate was formed as Schiff base complex compounds and were filtered off and dried in vacuo over

Table 1. Elemental analysis of the ligand (Schiff base) and the complexes.

Ligand and Complexes	% Carbon		% Hydrogen		% Nitrogen	
	Calculated	Found	Calculated	Found	Calculated	Found
SB (C ₁₄ H ₁₁ NO ₃) yellow powdered	69.70	69.62	4.56	4.44	5.80	5.78
[Ni (C ₁₄ H ₁₁ NO ₃) ₂]	62.14	61.66	4.06	3.85	5.17	4.54
[Cu (C ₁₄ H ₁₁ NO ₃) ₂]	61.59	61.02	4.03	3.64	5.13	4.70
[Co (C ₁₄ H ₁₁ NO ₃) ₂]	62.11	61.32	4.06	3.88	5.17	4.92
[Cd (C ₁₄ H ₁₁ NO ₃) ₂]	56.52	55.96	3.70	3.12	4.71	4.20
[Zn (C ₁₄ H ₁₁ NO ₃) ₂]	61.38	60.45	4.01	3.74	5.11	4.88
[Cr (C ₁₄ H ₁₁ NO ₃) ₃]	65.03	64.10	4.25	4.02	5.41	5.18

Where, SB = Schiff base.

Table 2. Physical properties of the ligand (Schiff base) and complexes.

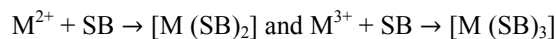
Complexes	Colour	Melting point or decomposition temp. (± 5°C)	% Yield	Molar conductance (ohm-2 cm ² mol ⁻¹)
[Ni (C ₁₄ H ₁₁ NO ₃) ₂]	Gray	207	63	6.5
[Cu (C ₁₄ H ₁₁ NO ₃) ₂]	Green	213	68	10.6
[Co (C ₁₄ H ₁₁ NO ₃) ₂]	Brown	300 (above)	66	6.7
[Cd (C ₁₄ H ₁₁ NO ₃) ₂]	Orange	270 (d)	64	11.9
[Zn (C ₁₄ H ₁₁ NO ₃) ₂]	Orange	300 (above)	59	22.9
[Cr (C ₁₄ H ₁₁ NO ₃) ₃]	Brown	300 (above)	61	9.3
SB: (C ₁₄ H ₁₁ NO ₃)	Yellow powdered	232	-	-

Where, SB = Schiff base

3.2. Infrared (IR) Spectral Studies

Infrared spectral data of the ligand and complexes were listed in Table 3. The spectral data of the ligands showed a strong absorption band at (1550-1650) cm⁻¹ due to ν (C=N) stretching [20-21] indicating the condensation have taken place between the CHO moiety of p- hydroxy benzaldehyde and -NH₂ moiety of 4-aminobenzoic acid. Band at (3020-3165) cm⁻¹ is responsible due to aromatic ν (C-H) stretching.

anhydrous CaCl₂. The general scheme of the complexes are mentioned as equation as;



Where, M²⁺ = Ni (II), Cu (II), Co (II), Cd (II) and Zn (II) ions, M³⁺ = Cr (III) ion, SB = Schiff base

A good yield of 60-70% was obtained and the purity of the complexes were confirmed by the elemental analysis.

3. Results and Discussion

All the complexes are stable at room temperature and are insoluble in common organic solvents but are soluble in DMSO, DMF and CHCl₃.

3.1. Elemental Analysis and Conductivity Measurement

The elemental analysis and physical properties of the complexes are listed in Table 1 and Table 2. The analytical data were in good agreement with the proposed empirical formula of the complexes. The conductance values of the complexes revealed that those were non-electrolytes in nature. [19]. The obtained values imply that no anions present outside the coordination sphere in all the complexes [28].

A band at (3300-3500) cm⁻¹ is liable due to ν (N-H) stretching of ν (NH₂) modes. The Band at (500-600) cm⁻¹ is accountable due to ν (M-N) stretching [22-23] which indicated the co-ordination through N atom to the metal. A band at (1600-1800) cm⁻¹ due to ν (C=O) stretching, band at (460-500) cm⁻¹ due to ν (M-O) stretching [24], and a band at (3400-3600) cm⁻¹ due to ν (O-H) stretching.

Table 3. IR spectral data of the ligand and metal complexes.

Complexes	ν (O-H) cm-1	ν (C = N) cm-1	ν (C-H) of aromatic cm-1	ν (M-O) cm-1	ν (M-N) cm-1	ν (C = O) cm-1
[Ni (C ₁₄ H ₁₁ NO ₃) ₂]	3412.24	1568.52	3050.10	495.93	534.39	1688.17
[Cu (C ₁₄ H ₁₁ NO ₃) ₂]	3404.20	1576.78	3070.87	426.54	527.05	1697.82
[Co (C ₁₄ H ₁₁ NO ₃) ₂]	3413.70	1568.76	3090.47	495.43	534.25	1683.02
[Cd (C ₁₄ H ₁₁ NO ₃) ₂]	3401.35	1569.91	3070.10	495.56	534.53	1686.03
[Zn (C ₁₄ H ₁₁ NO ₃) ₂]	3402.10	1570.50	3098.10	495.41	534.28	1688.62
[Cr (C ₁₄ H ₁₁ NO ₃) ₃]	3449.89	1575.17	3071	484.25	527.07	1693.04

3.3. Magnetic Moment and Electronic Spectra

The observed magnetic moment values of the complexes at ambient temperature are given in Table-4. The magnetic moment value of Ni (II) complex is diamagnetic which indicating the absence of unpaired electron. It appears from the magnetic moment data that complex is a low spin Square planar [25] complex. The observed magnetic moment value of Cu (II) complex was found to be 2.21 B. M. corresponding to one unpaired electron. It appears from the magnetic moment data that complex is a high spin tetrahedral complex. The observed magnetic moment value of Co (II) complex was observed 1.96 B. M. corresponding to one unpaired electron. It appears from the magnetic moment data that complex is also a high spin Square planar complex. The observed magnetic moment value of Cd (II) complex is diamagnetic indicating the absence of unpaired electron. It

appears from the magnetic moment data that complex is a low spin tetrahedral complex. The observed magnetic moment value of Zn (II) complex is diamagnetic indicating the absence of unpaired electron. It appears from the magnetic moment data that complex is a low spin tetrahedral complex. The observed magnetic moment value of Cr (III) complex is 3.89 B. M. corresponding to three unpaired electrons. It appears from the magnetic moment data that complex is a high spin octahedral [21] complex.

The electronic spectral data of the complexes are shown in Table 5. The electronic spectrum for the complexes are 298 nm, 297 nm, 336 nm 300 nm and 302 nm. These electronic spectra show bands in the region 200-420 nm due to charge transfer only [25]. The UV-visible spectrum of the complex of Cr (III) show an absorption band at 430 nm which is assigned the complex in an octahedral geometry [26].

Table 4. Magnetic moment data of the complexes.

Complexes	Length of the sample, 'l' in cm	weight of the sample 'm' in g	Susceptibility of the empty tube, Ro	Susceptibility of the sample with tube. R	Mass Susceptibility $\chi_g \times 10^{-6}$ C.G.S unit
[Ni (C ₁₄ H ₁₁ NO ₃) ₂]	2.3	0.0602	-61	-68	-0.557
[Cu (C ₁₄ H ₁₁ NO ₃) ₂]	2.1	0.0457	-67	-33	3.259
[Co (C ₁₄ H ₁₁ NO ₃) ₂]	2.8	0.0593	-68	-43	2.46
[Cd (C ₁₄ H ₁₁ NO ₃) ₂]	2.5	0.0784	-41	-60	-1.2638
[Zn (C ₁₄ H ₁₁ NO ₃) ₂]	2.5	.0599	-68	-72	-0.870
[Cr (C ₁₄ H ₁₁ NO ₃) ₃]	2.1	.0212	-66	-30	7.79

Table 4. Continued.

Complexes	Molecular weight of the sample, M	Molecular susceptibility $\chi_m \times 10^{-6}$ C.G.S. unit	Dimagnetic correction $\chi_L \times 10^{-6}$ C.G.S. unit	$\chi_{mcorr} \times 10^{-6}$ C.G.S. unit	μ_{eff} in B. M.
[Ni (C ₁₄ H ₁₁ NO ₃) ₂]	540.69	-301.16	-270.84	-30.32	dia
[Cu (C ₁₄ H ₁₁ NO ₃) ₂]	545.54	1777.91	-270.84	2048.75	2.21
[Co (C ₁₄ H ₁₁ NO ₃) ₂]	540.93	1330.68	-270.84	1601.52	1.96
[Cd (C ₁₄ H ₁₁ NO ₃) ₂]	594.41	-751.21	-272.84	-487.37	dia
[Zn (C ₁₄ H ₁₁ NO ₃) ₂]	547.39	-476.23	-272.84	-203.39	dia
[Cr (C ₁₄ H ₁₁ NO ₃) ₃]	774.99	6037.17	-273.84	6301.01	3.89

Table 5. Electronic spectral data of complexes.

Complexes	λ_{max} (nm)
[Ni (C ₁₄ H ₁₁ NO ₃) ₂]	298
[Cu (C ₁₄ H ₁₁ NO ₃) ₂]	297
[Co (C ₁₄ H ₁₁ NO ₃) ₂]	336
[Cd (C ₁₄ H ₁₁ NO ₃) ₂]	300
[Zn (C ₁₄ H ₁₁ NO ₃) ₂]	302
[Cr (C ₁₄ H ₁₁ NO ₃) ₃]	304, 430

4. Structure of the Complexes

From the above discussion (elemental analysis, conductivity measurement, magnetic moment and electronic spectra, IR spectra) and the literature review, it can be concluded that the possible structures of the complexes are given below:

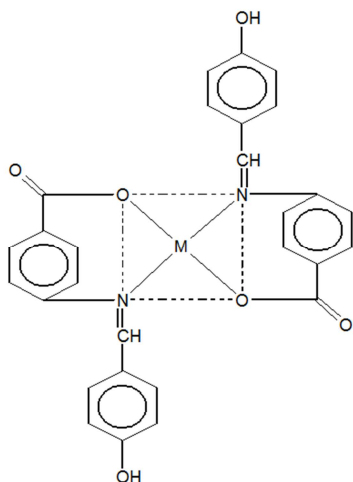


Figure 3. Probable structure of the complexes, Here, $M = \text{Ni (II)}$ and Co(II) .

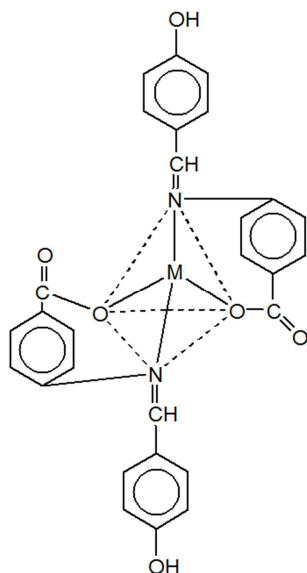


Figure 4. Probable structure of the complexes, Here, $M = \text{Cu (II)}$, Cd (II) and Zn (II) .

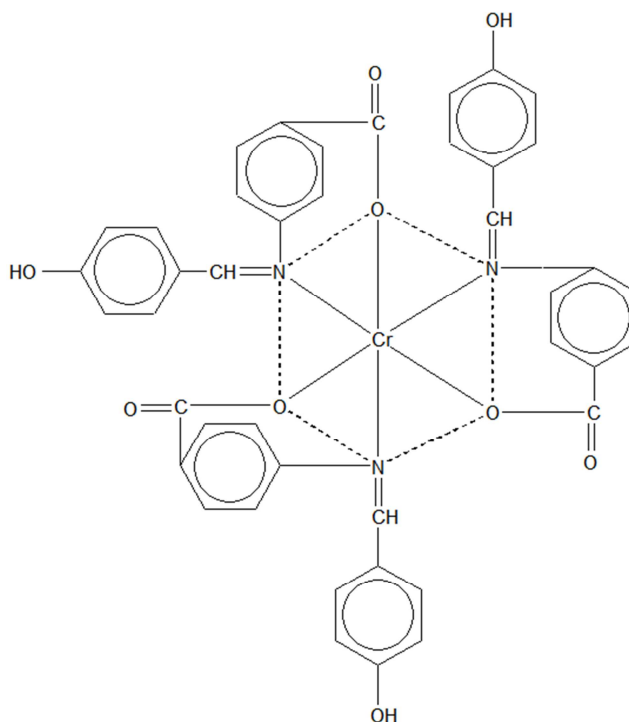


Figure 5. Probable structure of Cr (III) complex.

5. Antibacterial Activity of the Metal Complexes

Antimicrobial screening activities of the test samples are expressed by measuring the zone of inhibition observed around the area as shown in Table 6. The results revealed that the complexes are more microbial toxic than the free metal ions or ligands. All the complexes of metals under investigations more as well as less activities against the four pathogenic (two gram-positive and two gram-negative) bacteria tested. The results also revealed that among all the tested samples, these metal complexes showed strong activity against both the Gram positive and Gram-negative bacteria.

Table 6. Antibacterial screening activity of the complexes against gram-positive and gram-negative bacteria.

Compounds	Zone of inhibition, diameter in mm			
	Gram-Negative		Gram Positive	
	Escherichia coli	Shigella dysenteriae	Bacillus cereus	Streptococcus agalactiae
$[\text{Ni} (\text{C}_{14}\text{H}_{11}\text{NO}_3)_2]$	9	15	13	12
$[\text{Cu} (\text{C}_{14}\text{H}_{11}\text{NO}_3)_2]$	14	11	12	13
$[\text{Co} (\text{C}_{14}\text{H}_{11}\text{NO}_3)_2]$	6	11	5	9
$[\text{Cd} (\text{C}_{14}\text{H}_{11}\text{NO}_3)_2]$	9	-	6	7
$[\text{Zn} (\text{C}_{14}\text{H}_{11}\text{NO}_3)_2]$	11	16	7	12
$[\text{Cr} (\text{C}_{14}\text{H}_{11}\text{NO}_3)_3]$	10	5	9	8

6. Conclusion

Magnetic susceptibility measurement indicated that the Ni

(II), Cd (II) and Zn (II) complexes with Schiff base are the diamagnetic and the rest of the complexes are paramagnetic in nature. The IR spectral data showed that all the metals are coordinated through N (N of $-\text{N} = \text{CH} -$ and O of $-\text{C} = \text{O}$).

COO group of Schiff base) and O atom of the Schiff base the ligand coordinate with the metal. The electronic spectral data were in conformity with the Ni (II) and Co (II) are square planar structure of the complexes, Cu (II), Cd (II) and Zn (II) tetrahedral structure of complexes and octahedral structure of complex of Cr (III). Based on these above results and facts, the structures of complexes have been proposed as shown in Figure 3, 4 and 5. The Cu (II) complex shows the highest (potent) antimicrobial activity against gram-positive and gram-negative pathogenic bacteria according to standard kanamycin and ampicillin., Ni (II) and Zn (II) has potent antimicrobial activity against all the gram-positive and gram-negative pathogenic bacteria and the rest Co (II), Cd (II) and Cr (II) complexes with Schiff base exhibit moderate to less antimicrobial activity compared to examined complexes.

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