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SYNTHESIS OF $(C_8H_{11}N)_2 \cdot Zn(OAc)_2$, $(C_8H_{11}N)_2 \cdot Cu(OAc)_2$, AND $(C_8H_{11}N)_2 \cdot CuCl_2$ COMPLEXES AND APPLICATION TO THE HENRY REACTION© 2011 L. Mei^{1*}, C. Jie¹, H.B. Yang¹, Z.S. Ming², Y. Hao², T.H. Ming¹¹Hefei University of Technology, Department of Chemical Engineering, Hefei, 230009, China²University of Science and Technology of China, Hefei, 230009, China

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$(C_8H_{11}N)_2 \cdot Zn(OAc)_2$ (**1a**), $(C_8H_{11}N)_2 \cdot Cu(OAc)_2$ (**1b**), and $(C_8H_{11}N)_2 \cdot CuCl_2$ (**1c**) complexes are synthesized by a simple one-pot method. The crystal structures of **1a**, **1b**, and **1c** are determined by X-ray crystallography. The complexes are also characterized by NMR, IR, MS, and elemental analysis and used as the catalysts applied to the Henry reaction; moderate to high yields are obtained at room temperature.

Keywords: $(C_8H_{11}N)_2 \cdot Zn(OAc)_2$, $(C_8H_{11}N)_2 \cdot Cu(OAc)_2$, and $(C_8H_{11}N)_2 \cdot CuCl_2$ complexes, crystal structures, catalysts, Henry reaction.

Zinc and copper organometallic complexes involving amine ligands are widely used as catalysts in organic reactions and polymerizations [1–26], as evidenced by the numerous reports published in this area. This indicates the importance of these complexes in organic and polymer chemistry, since many groups have developed numerous organometallic polymerization catalysts and achieved satisfactory results [7–8, 13, 14]. In particular, zinc and copper acetate organometallic complexes have demonstrated good catalytic activity in the Henry reaction [18–26]. Inspired by their work, we synthesized novel $(C_8H_{11}N)_2 \cdot Zn(OAc)_2$ (**1a**), $(C_8H_{11}N)_2 \cdot Cu(OAc)_2$ (**1b**), and $(C_8H_{11}N)_2 \cdot CuCl_2$ (**1c**) complexes via a simple one-pot method. The crystalline forms of **1a**, **1b**, and **1c** were obtained at a low temperature or by slow evaporation at room temperature, and then characterized by NMR, IR, MS, elemental analysis, and X-ray analysis.

EXPERIMENTAL

Preparation of bis(α -ethylphenyl amine) zinc acetate complex (catalyst **1a).** Under water- and oxygen-free conditions, 2.7 ml (21.0 mmol) of α -ethylphenyl amine and 2.195 g (10.0 mmol) of $Zn(OAc)_2 \cdot 2H_2O$ were added in a dry 100-ml Schlenk flask. They were dissolved in 30 ml of dry ethanol and refluxed for 24 h. The solvent was filtrated, and recrystallization with petroleum ether/ethanol gave 1.68 g of white crystals; a yield of 39.4%. ¹H NMR (300 MHz, CDCl₃, 27 °C), ¹H NMR (500 MHz, CDCl₃) 7.22 ~ 7.31(m, 10H), 4.06 ~ 4.07 (d, *J* = 6.5 Hz, 1H), 3.06 (br, 1H), 1.94 (s, 6H), 1.40 ~ 1.42 (d, *J* = 6.5 Hz, 6H). ¹³C NMR (75 MHz, CDCl₃) 179.70, 144.04, 128.94, 127.92, 125.92, 51.82, 23.94, 23.10. IR (KBr), cm⁻¹: 3130, 1620, 1390, 1170, 702. HRMS (EI) *m/z*, %: Anal calcd for C₂₀H₂₈N₂O₄Zn: 424.1341; found: 424.1354, Elemental analysis, found: N: 6.45 %, C: 56.17 %, H: 6.56 %; Calculated: N: 6.61 %, C: 56.63 %, H: 6.62 %.

Preparation of bis(α -ethylphenyl amine) copper acetate complex (catalyst **1b).** Under water- and oxygen-free conditions, 2.7 ml (21.0 mmol) of α -ethylphenyl amine and 1.9965 g (20.0 mmol) of $Cu(OAc)_2 \cdot H_2O$ were added in a dry 100-ml Schlenk flask. They were dissolved in 15 ml of dry ace-

* E-mail: luomei@pku.edu.cn

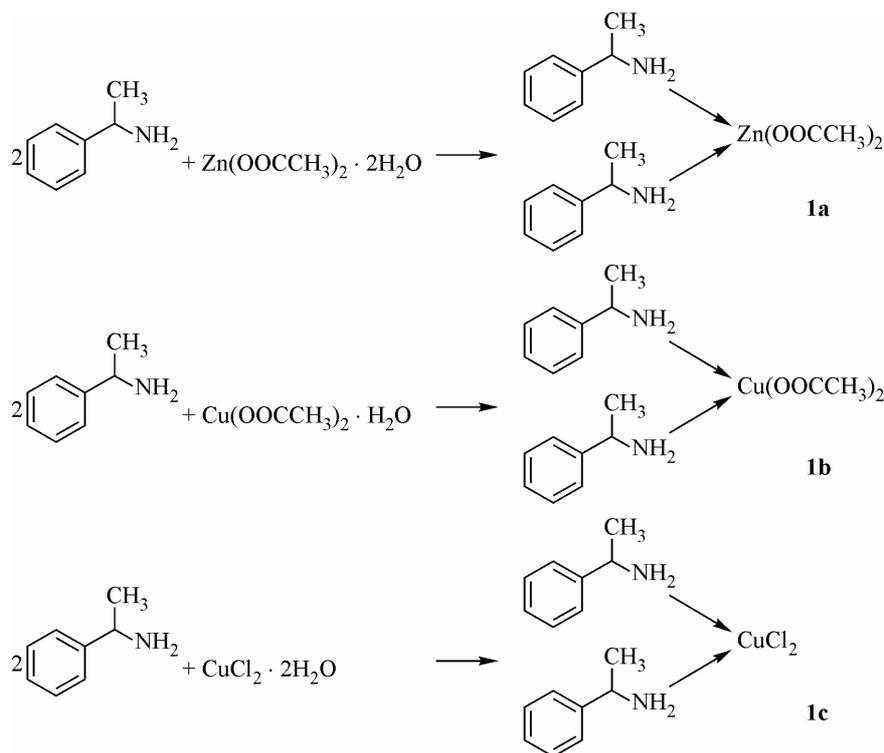


Fig. 1. Synthesis schemes of complexes **1a**, **1b**, **1c**

tonitrile and refluxed for 24 h. The solvent was reduced in vacuum, and recrystallization from a petroleum ether saturated solution gave 1.5 g of dark green crystals; yield 52.5 %. IR (KBr), cm^{-1} : 3220, 2970, 2350, 1560, 1390, 1160, 1090, 760, 702. Elemental analysis: found: N: 6.65 %, C: 56.42 %, H: 6.67 %; Calculated: N: 6.61 %, C: 56.65 %, H: 6.62 %.

Preparation of bis(α -ethylphenyl amine) cupric chloride complex (catalyst **1c).** Under water- and oxygen-free conditions, 2.7 ml (21.0 mmol) of α -ethylphenyl amine and 1.7048 g (10.0 mmol) of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ were added in a dry 100-ml Schlenk flask. They were dissolved in 15 ml of dry ethanol and refluxed for 24 h. The solvent was reduced in vacuum, the precipitation afforded a green solid; slow evaporation of saturated ethanol gave 0.88 g of green crystals; yield 51.6 %. IR (KBr), cm^{-1} : 3319, 3232, 3149, 2975, 2928, 1593, 1580, 1496, 1452, 1388., 1375, 1228, 1163, 1195, 1134, 1077, 1027, 1002, 988, 892, 775, 760. HRMS (EI) m/z , %: Anal calcd for $\text{C}_{16}\text{H}_{22}\text{N}_2\text{Cl}_2\text{Cu}$: 375.0456; found: 375.0458, Elemental analysis, found: N: 7.23 %; C: 50.58 %, H: 5.98 %; Calculated: N: 7.43 %, C: 50.58 %, H: 5.88 %.

2-Nitro-1-phenylethanol. Catalysts **1a**, **1b** or **1c** (0.59 mmol), benzaldehyde (0.20 ml, 1.97 mmol), and nitromethane (1.0 ml, 18 mmol) were successively mixed at room temperature. After 72 h, the reaction was quenched. Further purification was performed by silica gel. The title compound was obtained as a colorless liquid. ^1H NMR (300 MHz, CDCl_3): 7.28 ~ 7.32 (m, 5H, Ar—H), 5.32 ~ 5.35 (d, $J = 9.18$ Hz, 1H, —CH), 4.38 ~ 4.56 (m, 2H, — CH_2), 3.89 (br, 1H, —OH). ^{13}C NMR (75 MHz, CDCl_3): 138.30 128.92($\times 2$), 128.82, 125.98($\times 2$), 81.16, 70.91. HRMS (EI) m/z , %: Anal calcd for $\text{C}_8\text{H}_9\text{NO}_3$: 167.0582; found: 167.0588.

RESULTS AND DISCUSSION

Synthesis of complexes. The α -ethylphenyl amine ligand was treated with $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$ in THF, and the desired complex was obtained after refluxing for 24 h. The crude complex was recrystallized from petroleum ether and ethanol, and a crystal of **1a** was obtained after three days (Fig. 2, a).

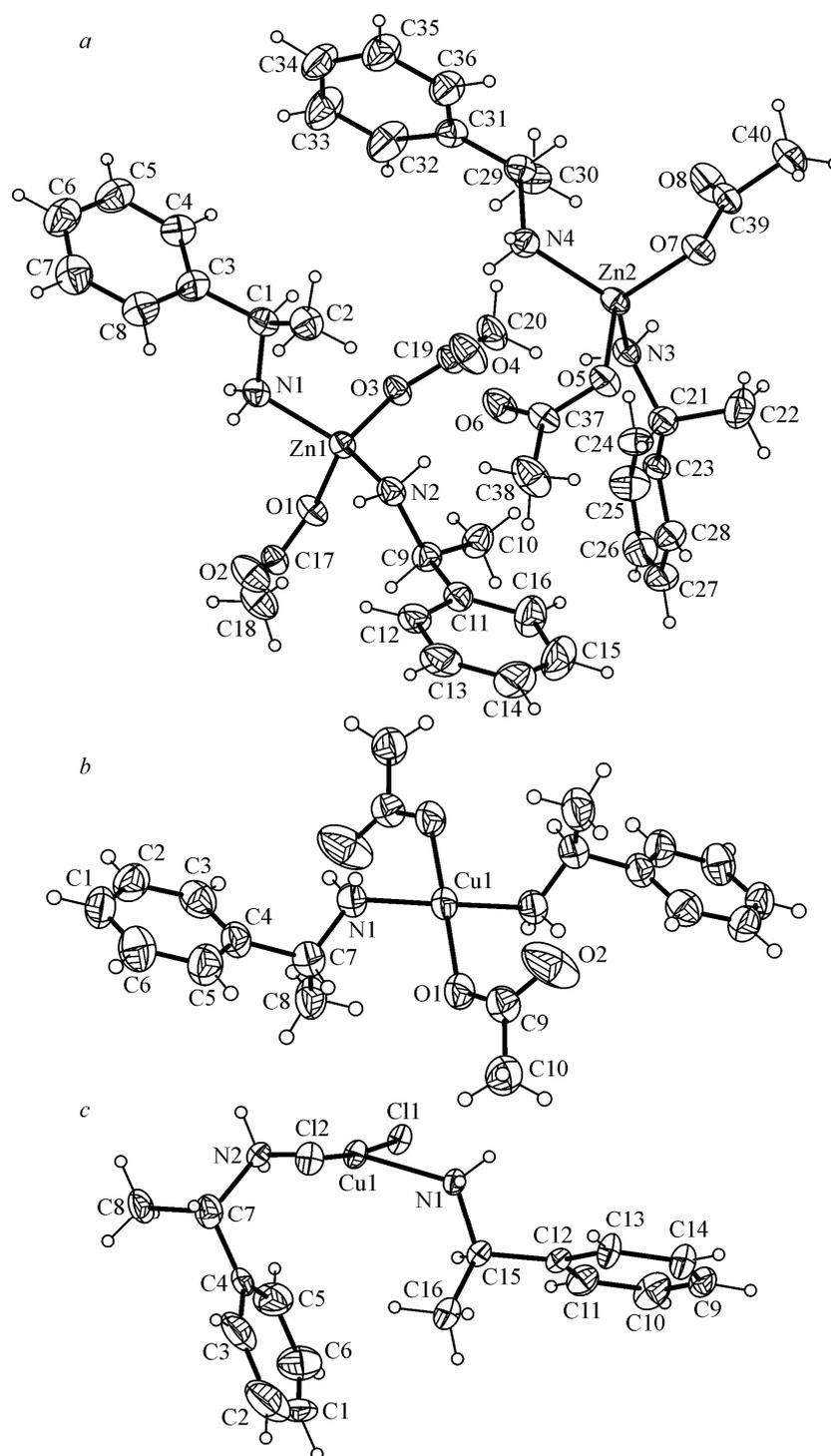


Fig. 2. Crystal structure of complex **1a**, **1b**, **1c**

By reacting the α -ethylphenyl amine ligand with $Cu(OAc)_2 \cdot H_2O$ in THF or ethanol and refluxing for 24 h, complex **1b** was obtained. It was then recrystallized from a saturated petroleum ether solvent (Fig. 2, *b*).

Complex **1c** was obtained from the reaction of α -ethylphenyl amine with $CuCl_2 \cdot 2H_2O$ in THF, and a pale blue solid was obtained after evaporation under vacuum. It easily dissolved in CH_2Cl_2 and

THF, but was poorly soluble in hexane and ethanol at room temperature. Two methods were attempted to obtain the crystals. The pale solid was separated by method I (recrystallization from petroleum and THF at $-30\text{ }^{\circ}\text{C}$) but a crystal of complex **1c** was afforded using method II: slow evaporation from ethanol.

Crystal structure. Structure determination. The structures of **1a**, **1b**, and **1c** were studied by X-ray diffraction (Fig. 2). Tables 1—3 list the selected bond lengths and angles for the three compounds.

A colorless block crystal of title compound **1a** with approximate dimensions of $0.31 \times 0.24 \times 0.16$ mm was selected for the data collection on a BRUKER SMART diffractometer with graphite monochromated MoK_{α} radiation ($\lambda = 0.7103\text{ \AA}$). A total of 6428 reflections were collected in the range of $3.39 < \theta < 63.36^{\circ}$ by using "phi and omega" scan techniques at 293(2) K. $\text{C}_{20}\text{H}_{28}\text{N}_2\text{O}_4\text{Zn}$, $M = 425.81$, monoclinic, $P2_1$, 21, 21, $a = 10.662(5)$, $b = 14.912(5)$, $c = 26.991(5)\text{ \AA}$, $\alpha = \beta = \gamma = 90^{\circ}$, $V = 1691\text{ \AA}^3$,

Table 1

Selected bond lengths d , \AA and bond angles ω , ged. for the N—Zn complex (catalyst **1a**)

Bond	d	Bond	d	Bond	d
Zn(2) ~ O(7)	1.9568(16)	O(8) ~ C(39)	1.224(3)	C(29) ~ C(31)	1.507(3)
Zn(2) ~ O(5)	1.9683(16)	N(2) ~ C(9)	1.486(3)	C(29) ~ C(30)	1.525(3)
Zn(2) ~ N(4)	2.0255(18)	N(3) ~ C(21)	1.480(3)	C(21) ~ C(23)	1.513(3)
Zn(2) ~ N(3)	2.0405(17)	O(4) ~ C(19)	1.220(3)	C(17) ~ C(18)	1.508(4)
Zn(1) ~ O(3)	1.9492(16)	O(7) ~ C(39)	1.278(3)	C(37) ~ C(38)	1.490(4)
Zn(1) ~ O(1)	1.9561(17)	O(1) ~ C(17)	1.267(3)	C(9) ~ C(10)	1.503(3)
Zn(1) ~ N(2)	2.0228(16)	O(2) ~ C(17)	1.220(3)	C(19) ~ C(20)	1.491(4)
Zn(1) ~ N(1)	2.0319(19)	N(1) ~ C(1)	1.486(3)	C(1) ~ C(2)	1.506(3)
O(3) ~ C(19)	1.281(3)	C(39) ~ C(40)	1.499(3)	C(1) ~ C(3)	1.518(4)
O(5) ~ C(37)	1.279(3)	C(29) ~ N(4)	1.483(3)	C(11) ~ C(9)	1.523(3)
O(6) ~ C(37)	1.223(3)				
Angle	ω	Angle	ω	Angle	ω
O(7) ~ Zn(2) ~ O(5)	98.61(7)	C(17) ~ O(1) ~ Zn(1)	124.19(15)	O(2) ~ C(17) ~ O(1)	124.0(2)
O(7) ~ Zn(2) ~ N(4)	110.28(8)	C(1) ~ N(1) ~ Zn(1)	118.49(15)	O(2) ~ C(17) ~ C(18)	121.2(2)
O(5) ~ Zn(2) ~ N(4)	105.45(7)	O(8) ~ C(39) ~ O(7)	123.4(2)	O(1) ~ C(17) ~ C(18)	114.8(2)
O(7) ~ Zn(2) ~ N(3)	121.55(7)	O(8) ~ C(39) ~ C(40)	121.1(2)	O(6) ~ C(37) ~ O(5)	123.8(2)
O(5) ~ Zn(2) ~ N(3)	104.22(7)	O(7) ~ C(39) ~ C(40)	115.5(2)	O(6) ~ C(37) ~ C(38)	121.1(2)
N(4) ~ Zn(2) ~ N(3)	113.96(8)	N(4) ~ C(29) ~ C(31)	110.8(2)	O(5) ~ C(37) ~ C(38)	115.0(2)
O(3) ~ Zn(1) ~ O(1)	97.56(7)	N(4) ~ C(29) ~ C(30)	108.23(19)	N(2) ~ C(9) ~ C(10)	109.3(2)
O(3) ~ Zn(1) ~ N(2)	118.06(7)	C(31) ~ C(29) ~ C(30)	115.1(2)	N(2) ~ C(9) ~ C(11)	111.98(18)
O(1) ~ Zn(1) ~ N(2)	110.95(8)	C(29) ~ N(4) ~ Zn(2)	121.55(15)	C(10) ~ C(9) ~ C(11)	114.8(2)
O(3) ~ Zn(1) ~ N(1)	111.36(8)	C(16) ~ C(11) ~ C(9)	122.8(3)	O(4) ~ C(19) ~ O(3)	123.4(2)
O(1) ~ Zn(1) ~ N1	106.25(8)	C(12) ~ C(11) ~ C(9)	119.2(2)	O(4) ~ C(19) ~ C(20)	121.3(2)
N(2) ~ Zn(1) ~ N1	111.29(8)	C(36) ~ C(31) ~ C(29)	120.2(2)	O(3) ~ C(19) ~ C(20)	115.3(2)
C(19) ~ O(3) ~ Zn(1)	120.88(14)	C(32) ~ C(31) ~ C(29)	123.9(2)	N(1) ~ C(1) ~ C(2)	109.4(2)
C(37) ~ O(5) ~ Zn(2)	114.56(15)	N(3) ~ C(21) ~ C(22)	108.5(2)	N(1) ~ C(1) ~ C(3)	111.1(2)
C(9) ~ N(2) ~ Zn(1)	113.92(13)	N(3) ~ C(21) ~ C(23)	115.04(19)	C(2) ~ C(1) ~ C(3)	112.9(2)
C(21) ~ N(3) ~ Zn(2)	112.25(13)	C(22) ~ C(21) ~ C(23)	111.7(2)	C(3) ~ C(8) ~ C(7)	121.5(3)
C(39) ~ O(7) ~ Zn(2)	118.85(14)				

$Z = 8$, $D_x = 1.318 \text{ g/cm}^3$, the final R factor was $R_1 = 0.0246$, 6045 for reflections with $I_0 > 2\sigma(I_0)$, $R\omega = 0.0658$ for all data. The structure was solved by full-matrix least-squares on F^2 using the SHELXTL program [26, 27].

A blue block monoclinic crystal of title compound **1b** of approximately $0.38 \times 0.20 \times 0.12 \text{ mm}$ was selected for the data collection on a BRUKER SMART diffractometer with graphite monochromated $\text{MoK}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). A total of 17573 reflections were collected in the range of $3.66 < \theta < 29.11^\circ$ by using "phi and omega" scan techniques at 293(2) K. $\text{C}_{20}\text{H}_{28}\text{N}_2\text{O}_4\text{Cu}$, $M = 423.98$, orthorhombic, $P-1$, $a = 5.472(5)$, $b = 9.225(5)$, $c = 11.094(5) \text{ \AA}$, $\alpha = 67.153$, $\beta = 85.263$, $\gamma = 82.016^\circ$, $V = 510.8 \text{ \AA}^3$, $Z = 1$, $D_x = 1.378 \text{ g/cm}^3$, the final R factor was $R_1 = 0.0455$, 1784 for reflections with $I_0 > 2\sigma(I_0)$, $R\omega = 0.0917$ for all data; the largest peak and hole were 0.641 and -0.635 e\AA^{-3} respectively. The structure was solved by full-matrix least-squares on F^2 using the the SHELXTL program [27, 28].

Table 2

Selected bond lengths d , \AA and bond angles ω , ged. for the N—Cu complex (catalyst **1b**)

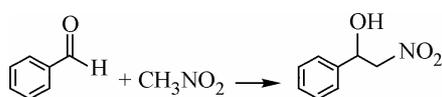
Bond	d	Bond	d	Bond	d
Cu(1) ~ O(1)	1.946(2)	O(2) ~ C(9)	1.223(4)	C(1) ~ C(2)	1.375(4)
Cu(1) ~ O(1)	1.946(2)	C(6) ~ C(1)	1.367(4)	C(3) ~ C(4)	1.381(4)
Cu(1) ~ N(1)	2.003(2)	C(6) ~ C(5)	1.378(4)	C(3) ~ C(2)	1.395(4)
Cu(1) ~ N(1)	2.003(2)	C(9) ~ C(10)	1.502(4)	C(4) ~ C(7)	1.531(4)
O(1) ~ C(9)	1.258(3)	C(5) ~ C(4)	1.368(4)	C(7) ~ C(8)	1.501(4)
N(1) ~ C(7)	1.466(4)				
Angle	ω	Angle	ω	Angle	ω
O(1) ~ Cu(1) ~ O(1)	180.000(1)	C(9) ~ O(1) ~ Cu(1)	123.6(2)	C(5) ~ C(4) ~ C(7)	120.0(3)
O(1) ~ Cu(1) ~ N(1)	90.26(10)	C(7) ~ N(1) ~ Cu(1)	123.19(19)	C(3) ~ C(4) ~ C(7)	121.7(3)
O(1) ~ Cu(1) ~ N(1)	89.74(10)	O(2) ~ C(9) ~ O(1)	123.9(3)	N(1) ~ C(7) ~ C(8)	110.8(3)
O(1) ~ Cu(1) ~ N(1)	89.74(10)	O(2) ~ C(9) ~ C(10)	119.1(3)	N(1) ~ C(7) ~ C(4)	111.5(2)
O(1) ~ Cu(1) ~ N(1)	90.26(10)	O(1) ~ C(9) ~ C(10)	116.7(3)	C(8) ~ C(7) ~ C(4)	112.1(2)
N(1) ~ Cu(1) ~ N(1)	180.000(1)				

Table 3

Selected bond lengths d , \AA and bond angles ω , ged. for the N—Cu complex (catalyst **1c**)

Bond	d	Bond	d	Bond	d
Cu(1) ~ N(2)	1.990(4)	Cu(1) ~ Cl(1)	2.3156(17)	C(4) ~ C(7)	1.500(6)
Cu(1) ~ N(1)	1.991(4)	N(1) ~ C(15)	1.508(6)	C(15) ~ C(16)	1.487(6)
Cu(1) ~ Cl(2)	2.3020(17)	N(2) ~ C(7)	1.485(6)	C(7) ~ C(8)	1.530(6)
Angle	ω	Angle	ω	Angle	ω
N(2) ~ Cu(1) ~ N(1)	164.75(19)	C(7) ~ N(2) ~ Cu(1)	118.3(3)	N(2) ~ C(7) ~ C(4)	111.8(4)
N(2) ~ Cu(1) ~ Cl(2)	91.47(13)	C(13) ~ C(12) ~ C(15)	122.8(5)	N(2) ~ C(7) ~ C(8)	112.1(4)
N(1) ~ Cu(1) ~ Cl(2)	91.59(11)	C(11) ~ C(12) ~ C(15)	118.8(5)	C(16) ~ C(15) ~ N(1)	109.8(4)
N(2) ~ Cu(1) ~ Cl(1)	89.77(13)	C(5) ~ C(4) ~ C(7)	121.6(6)	C(16) ~ C(15) ~ C(12)	112.4(4)
N(1) ~ Cu(1) ~ Cl(1)	88.65(11)	C(3) ~ C(4) ~ C(7)	118.9(7)	N(1) ~ C(15) ~ C(12)	113.2(4)
Cl(2) ~ Cu(1) ~ Cl(1)	174.36(8)	C(16) ~ C(15) ~ N(1)	109.8(4)	C(4) ~ C(7) ~ C(8)	111.5(5)
C(15) ~ N(1) ~ Cu(1)	114.6(3)				

Table 4

Effect of catalysts^a

Entry	Catalyst	Time(h)	Conv. (%) ^b
1	1a	72	>99
2	1b	72	82
3	1c	72	63
4	no	72	<5

^a Reactions were carried out on 0.2 ml (1 mmol) PhCHO with 1 ml CH₃NO₂ in 4 ml CH₃OH, 72 h, at room temperature (20–30°C).

^b The conv. % was given by ¹H NMR.

A blue block triclinic crystal of title compound **1c** of approximately 0.38×0.20×0.12 mm was selected for the data collection on a BRUKER SMART diffractometer with graphite monochromated MoK_α radiation ($\lambda = 0.71073 \text{ \AA}$). A total of 5043 reflections were collected in the range of $2.9 < \theta < 28.82^\circ$ using "phi and omega" scan techniques at 293(2) K, C₃₂H₄₄N₄Cl₄Cu₂, $M = 753.59$, orthorhombic, $P-1$, $a = 6.461(5) \text{ \AA}$, $\alpha = 111.056(5)^\circ$, $b = 12.017(5)$, $\beta = 95.542^\circ$, $c = 12.368(5) \text{ \AA}$, $\gamma = 90.559^\circ$, $V = 890.9(9) \text{ \AA}^3$, $Z = 1$, $D_x = 1.405 \text{ g/cm}^3$, the final R factor was $R_1 = 0.0506$, 1524 for reflections with $I_0 > 2\sigma(I_0)$, $R_w = 0.0667$ for all data. The structure was solved by full-matrix least-squares on F^2 using the SHELXTL program [27, 28].

Complexes **1a** and **1b** were each composed of two molecules of α -ethylphenyl amine and a Zn(Ac)₂ core, and Cu(Ac)₂ respectively (Fig. 2, *a, b*). For complex **1a**, the bond length of N(2) ~ Zn(1) was 2.0228(16) Å,

shorter than that of N(1) ~ Zn(1) of 2.0319(19) Å. The chirality around a zinc ion was N(1), N(2), O(1), and O(3), with these atoms forming a tetrahedron with a zinc ion in the center. The bond angle of N ~ Zn ~ O was nearly 109.5°. Additionally, the presence of hydrogen bonds should be mentioned. Four α -ethylphenyl amine molecules and two Zn(OAc)₂ cores were connected together to construct a hydrogen-bonded dinuclear structure (Fig. 2, *a*). Unlike **1a**, complex **2** is centrosymmetric around the Cu(II) cation having tetragonal geometry and site symmetry $\bar{1}$, coordinated by the two N atoms from the ligating α -ethylphenyl amine and by the two acetate anion.

Complex **1c** was composed of four molecules of α -ethylphenyl amine and two CuCl₂ cores (Fig. 2, *c*). The geometry at the Cu ion contained two Cl⁻ ions and two N atoms from α -ethylphenyl amine, in an approximately tetragonal shape. The four N—Cu—Cl angles were all approximately 90°, and two Cl—Cu bond lengths (2.3020, 2.3156 Å) were longer than two N—Cu bond lengths (1.990, 1.994 Å).

Application to the Henry reaction. Complexes **1a**, **1b**, and **1c** were used as the catalysts in application to the Henry reaction at room temperature. After 72 hours, catalyst **1a** showed the best catalytic activity and the conversion rate of almost 100 %, with catalysts **1b** and **1c** also showing good catalytic effect (Table 4), but no catalytic effect was observed in the absence of catalysts.

The XRD pattern has not showed a significant change after the reaction and the same did the HRMS analysis of catalysts after the reaction. These data further show that the complex is a catalyst involved in the catalytic reaction.

CONCLUSIONS

In summary, we have synthesized N—Zn, N—Cu complexes bearing α -ethylphenyl amine and obtained their crystal structures. The novelty of this methodology can be summarized as follows: (1) synthesis of a series of catalysts by a simple one-step method from inexpensive and readily available raw materials (the ligand: copper acetate, zinc acetate. and cupric chloride), and (2) first reported of novel catalysts of the Henry reaction at room temperature with moderate to high yields. Efforts will be devoted to further improve the conversion rate of benzaldehyde, to study the response of other aldehydes, and will be applied to other organic reactions such as cyanosilylation and aldol reactions, etc.

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