Scientific paper

Synthesis, Characterization and Crystal Structures of Nitrate and Tetrachloridocobalt(II) Salts of Cobalt(III) Complexes with Catalytic Epoxidation of Cyclooctene

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Received: 05-08-2025

Abstract

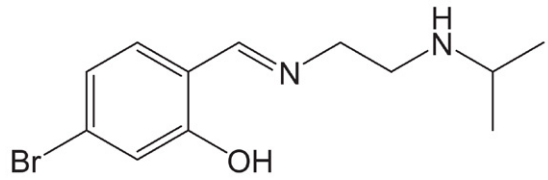
Two new cobalt(III) complexes, $[CoL_2]NO_3$ (**1**) and $[CoL_2]_2[CoCl_4]$ (**2**), where L is 5-bromo-2-(((2-isopropylamino) ethyl)imino)methyl)phenolate, have been prepared and characterized by physico-chemical methods and single crystal X-ray analysis. X-ray analysis indicated that the Co atoms in both complexes are in octahedral coordination except for that in $[CoCl_4]$ unit which in a tetrahedral coordination. Crystal structures of complexes are stabilized by hydrogen bonds and π···π interactions. Catalytic property of both cobalt complexes was studied on the epoxidation of cyclooctene with *tert*-butylhydroperoxide (TBHP) as oxidant. Both complexes show good catalytic activity and high epoxides selectivity.

Keywords: Schiff base; Cobalt complex; Crystal structure; Catalytic property

1. Introduction

Schiff bases are readily synthesized by condensation reaction of carbonyl compounds with primary amines. Schiff bases have been widely investigated for their biological activities, such as antibacterial and antitumor activities.[1] Metal complexes of Schiff bases have also been received much attention. These complexes show interesting properties in catalytic and enzymatic reactions, magnetism and molecular architectures,[2] as well as biological activities.[3]

In the last years, Schiff base complexes have been widely used as catalysts for sulfoxidation,[4] epoxidation,[5] etc. Among the complexes, those with cobalt center have received particular attention in the field of catalytic epoxidation reaction.[6] The oxygen binding ability of cobalt complexes stimulated the study on reversible oxygen binding of cobalt complexes and their usage as catalysts.[7] Epoxides are important precursors for the production of a variety of fine chemicals. Thus, the catalytic epoxidation of alkenes is an important reaction. A number of cobalt complexes have shown remarkable and interesting catalytic properties for the epoxidation of olefins.[8] During search of literature, cobalt complexes with Schiff bases derived from 4-bromosalicylaldehyde are seldom reported. As continuation of this work to explore more efficient new catalysts, we report herein the synthesis, characterization, crystal structures, and catalytic properties of two new cobalt(III) complexes, $[CoL_2]NO_3$ (**1**) and $[CoL_2]_2[CoCl_4]$ (**2**), where L is 5-bromo-2-(((2-isopropylamino)ethyl)imino)methyl) phenolate (HL; Scheme 1).



Scheme 1. The Schiff base HL.

2. Experimental

2. 1. Materials and Measurements

4-Bromosalicylaldehyde and *N*-isopropylethane-1,2-diamine with AR grade were obtained from Aldrich

and used as received. Cobalt nitrate and cobalt chloride were purchased from TCI. Elemental analyses were performed using a Perkin-Elmer 240C analytical instrument. Infrared spectra were recorded on a Nicolet 5DX FT-IR spectrophotometer with KBr pellets. Molar conductance was measured with a Shanghai DDS-11A conductometer.

2. 2. Synthesis of $[CoL_2]NO_3$ (1)

4-Bromosalicylaldehyde (0.20 g, 1.0 mmol) and *N*-isopropylethane-1,2-diamine (0.10 g, 1.0 mmol) were dissolved in methanol (30 mL). To the mixture was added cobalt nitrate hexahydrate (0.29 g, 1.0 mmol). A brown solution was formed immediately. After 20 min stirring, the solution was filtered and the filtrate was kept for slow evaporation. The diffraction quality colorless single crystals that deposited over a period of a few days were collected by filtration and washed with methanol. The yield was 0.13 g (38%). Anal. Calcd. for $C_{24}H_{32}Br_2CoN_5O_5$ (%): C, 41.82; H, 4.68; N, 10.16. Found: C, 41.66; H, 4.61; N, 10.33. IR data (KBr, cm^{-1}): 3164, 1647, 1585, 1521, 1466, 1450, 1408, 1385, 1344, 1323, 1288, 1245, 1203, 1175, 1139, 1084, 1062, 1031, 977, 935, 913, 853, 798, 790, 730, 678, 620, 600, 561, 470. UV-Vis data in methanol [λ_{max} (nm), ε (L mol^{-1} cm^{-1})]: 225, 15370; 257, 18210; 378, 1923.

2. 3. Synthesis of $[CoL_2]_2[CoCl_4]$ (2)

4-Bromosalicylaldehyde (0.20 g, 1.0 mmol) and *N*-isopropylethane-1,2-diamine (0.10 g, 1.0 mmol) were dissolved in methanol (30 mL). To the mixture was added cobalt chloride hexahydrate (0.24 g, 1.0 mmol). A brown solution was formed immediately. After 20 min stirring, the solution was filtered and the filtrate was kept for slow evaporation. The diffraction quality colorless single crystals that deposited over a period of a few days were collected by filtration and washed with methanol. The yield was 0.22 g (30%). Anal. Calcd. for $C_{48}H_{64}Br_4Cl_4Co_3N_8O_4$ (%): C, 39.61; H, 4.43; N, 7.70. Found: C, 39.45; H, 4.53; N, 7.76. IR data (KBr, cm^{-1}): 3177, 1647, 1585, 1523, 1465, 1450, 1410, 1345, 1326, 1291, 1245, 1203, 1175, 1141, 1082, 1062, 1027, 975, 935, 911, 853, 798, 787, 722, 667, 635, 593, 560, 543, 475. UV-Vis data in methanol [λ_{max} (nm), ε (L mol^{-1} cm^{-1})]: 225, 16120; 260, 17870; 380, 2105.

2. 4. X-Ray Crystallography

Suitable single crystals of the complexes were selected and mounted on a Bruker Smart 1000 CCD area-detector diffractometer with graphite monochromatized Mo-Ka radiation (l = 0.71073 Å). Diffraction data for the compounds were collected by ω scan mode at 298(2) K. Data reduction and cell refinement were performed by the SMART and SAINT programs.[9] Empirical absorption correction was applied by using SADABS.[10] The structures were solved by direct methods and refined with the full-matrix least-squares technique using SHELXL97.[11] The non-H atoms in the structures were subjected to refined anisotropic refinement. The hydrogen atoms were located in geometrically and treated with the riding mode. Crystallographic data and experimental details for the compounds are summarized in Table 1. Selected bond lengths and angles for the compounds are listed in Table 2.

2. 5. Catalytic Oxidation Reaction

The two cobalt complexes were tested as catalysts for the oxidation of cyclooctene with TBHP as oxidant. The reaction was monitored by gas chromatography at 60 min

Table 1. Crystallographic data for the two cobalt complexes

	1	**2**
Molecular formula	$C_{24}H_{32}Br_2CoN_5O_5$	$C_{48}H_{64}Br_4Cl_4Co_3N_8O_4$
Molecular weight	689.30	1455.30
Crystal system	Monoclinic	Orthorhombic
Space group	*C*2/*c*	*Fddd*
a, Å	21.4695(15)	16.4270(17)
b, Å	12.7142(13)	25.7287(18)
c, Å	12.4588(13)	27.0688(18)
α, °	90	90
β, °	125.178(1)	90
γ, °	90	90
V, Å^3	2779.7(5)	11440.5(16)
Z	4	8
ρ_{calcd}, g cm^{-3}	1.647	1.690
μ, mm^{-1}	3.535	3.889
Reflections collected/unique	14875/2589	14744/2676
Observed reflections ($I \geq 2\sigma(I)$)	2229	1902
Data/restraints/parameters	2589/0/171	2676/0/164
GOOF on F^2	1.048	1.011
R_1, wR_2 ($I \geq 2\sigma(I)$)	0.0289, 0.0718	0.0444, 0.1019
R_1, wR_2 (all data)	0.0367, 0.0761	0.0699, 0.1166

Table 2. Selected bond distances (Å) and angles (°) for the two cobalt complexes

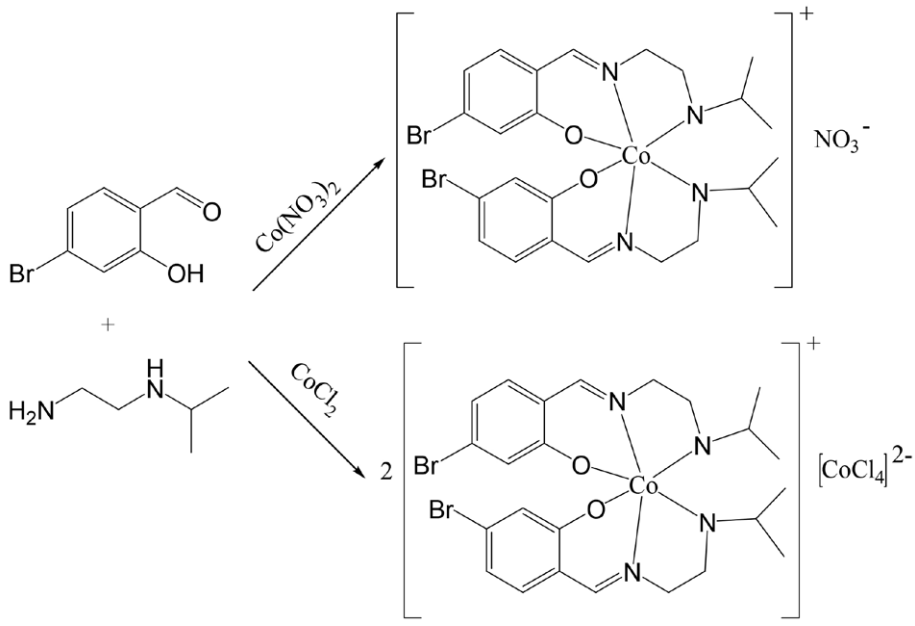
1			
Co(1)–O(1)	1.8970(17)	Co(1)–N(1)	1.905(2)
Co(1)–N(2)	2.034(2)		
O(1)–Co(1)–O(1A)	87.74(11)	O(1)–Co(1)–N(1A)	86.65(8)
O(1)–Co(1)–N(1)	94.29(8)	N(1)–Co(1)–N(1A)	178.69(13)
O(1)–Co(1)–N(2A)	90.39(8)	N(1)–Co(1)–N(2A)	94.46(8)
O(1)–Co(1)–N(2)	177.89(8)	N(1)–Co(1)–N(2)	84.62(8)
N(2)–Co(1)–N(2A)	91.50(12)		
Symmetry code: A) 1 – *x*, –*y*, –*z*.			
2			
Co(1)–O(1)	1.891(3)	Co(1)–N(1)	1.911(4)
Co(1)–N(2)	2.030(4)		
O(1)–Co(1)–O(1B)	88.93(18)	O(1)–Co(1)–N(1B)	84.55(14)
O(1)–Co(1)–N(1)	92.88(14)	N(1)–Co(1)–N(1B)	176.4(2)
O(1)–Co(1)–N(2B)	90.32(13)	O(1)–Co(1)–N(2)	177.38(13)
N(1)–Co(1)–N(2)	84.55(15)	N(1)–Co(1)–N(2B)	97.99(15)
N(2)–Co(1)–N(2B)	90.5(2)		
Cl(1)–Co(2)–Cl(1B)	106.50(8)	Cl(1)–Co(2)–Cl(1C)	109.95(11)
Cl(1)–Co(2)–Cl(1D)	112.01(11)		
Symmetry codes: B) 5/4 – *x*, *y*, 5/4 – *z*; C) 5/4 – *x*, 5/4 – *y*, *z*; D) *x*, 5/4 – *y*, 5/4 – *z*.			

intervals in different solvents and conditions. The retention times for the starting materials and the products were determined by comparison with authentic samples. In the absence of the catalysts, very little products were formed. Cyclooctеneoxide is the major product of the oxidation process with cyclooctene as the starting material. The catalyst (10 μmol) was dissolved in solvent (10 mL) and cyclooctene (15 mmol) and TBHP (30 mmol) were added. The mixture was stirred under reflux and with the reaction monitored at 60 min intervals by gas chromatography.

3. Results and Discussion

3. 1. Chemistry

Complexes **1** and **2** were prepared by *in situ* reaction of 4-bromosalicylaldehyde, *N*-isopropylethane-1,2-diamine, with cobalt nitrate and cobalt chloride, respectively in methanol (Scheme 2). As usually observed for the preparation of cobalt complexes, Co^{II} in $[CoL_2]$ units underwent aerial oxidation to Co^{III} in the synthetic route of both complexes. The molar conductivities of the complex-



Scheme 2. The synthetic route for both complexes.

es **1** and **2** measured in methanol at concentration of 10^{-3} mol L^{-1} are 223 and 345 Ω^{-1} cm^2 mol^{-1}, indicating the 1:1 and 1:2 electrolytic nature of the complexes in solution.[12]

3. 2. Infrared and Electronic Spectra

In the infrared spectra of the complexes, the weak absorptions in the range 3164–3177 cm^{-1} are assigned to the N–H vibrations of the Schiff base ligands. The characteristic imine stretching is observed at 1647 cm^{-1} as strong signal.[13] The spectrum of complex **1** shows an intense band at 1385 cm^{-1} characteristic of ionic nitrate.[14] The Schiff base ligands coordination is substantiated by the phenolic C–O stretching bands at 1175 cm^{-1} in the complexes.[15] Coordination of the Schiff bases is further confirmed by the appearance of weak bands in the low wave numbers 400–600 cm^{-1}, corresponding to ν(Co–N) and ν(Co–O).[16]

In the UV-Vis spectra of the complexes, the bands at 225 nm and 257–260 nm are attributed to the π-π^* and n-π^* transitions.[17] The bands at 378–380 nm can be attributed to the ligand to metal charge transfer transition (LMCT).[18]

3. 3. Crystal Structure Description of the Complexes

Molecular structures of complexes **1** and **2** are shown in Figs. 1 and 2, respectively. Complex **1** contains a $[CoL_2]^+$ cation and a nitrate anion, and complex **2** contains two $[CoL_2]^+$ cations and a $[CoCl_4]^{2-}$ anion. The Co atom in each $[CoL_2]^+$ cation is coordinated by two phenolate oxygen, two imino nitrogen and two amino nitrogen from two Schiff base ligands, forming octahedral coordination. The equatorial plane of the octahedral coordination is defined by O(1), N(1), N(2) and N(1A) atoms, and the axial positions are occupied by O(1A) and N(2A) atoms. The octahedral geometry is distorted from ideal model, as evidenced by the bond angles. The *cis* and *trans* angles in the octahedral coordination are in the ranges of 84.62(8)–94.46(8)° and 177.89(8)–178.69(8)° for **1**, and 84.55(15)–97.99(15)° and 176.4(2)–177.38(13)° for **2**, respectively. The Co–O and Co–N bond lengths are comparable to those observed in Schiff base cobalt complexes.[19] The Co atom in the $[CoCl_4]^{2-}$ anion is in a tetrahedral coordination, with the bond angles ranging from 106.50(8) to 112.01(11)°.

As shown in Fig. 3, the nitrate anions in complex **1** are linked to the complex cations through N–H···O hydrogen bonds (Table 3), to form a dimer. In addition, there are π···π interactions among the molecules (Cg(3)···Cg(3)^b 4.320(5) Å, symmetry code: b) $-x$, 1 – y, 1 – z; Cg(3) is the centroid of C(1)–C(2)–C(3)–C(4)–C(5)–C(6)). As shown in Fig. 4, the $[CoCl_4]^{2-}$ anions and the complex cations in complex **2** are linked through N–H···Cl and C–H···Cl hydrogen bonds (Table 3), to form two-dimensional sheets parallel to the *bc* plane. In addition, there are π···π interac-

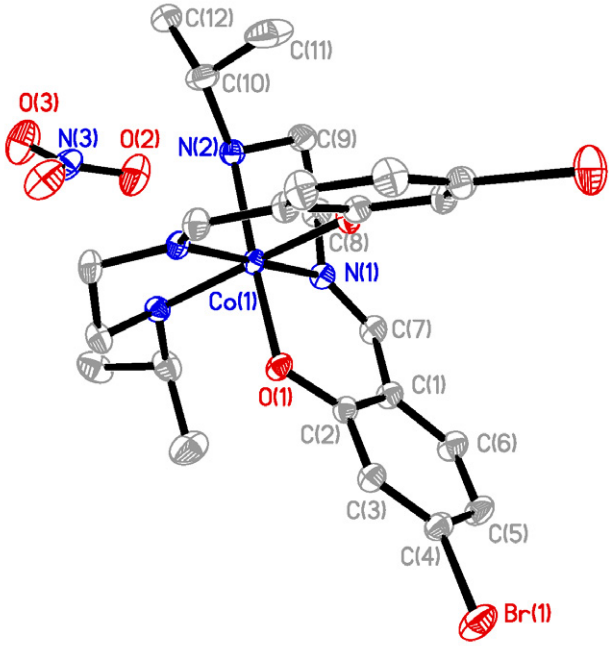


Fig. 1. Molecular structure of complex **1**. Unlabeled atoms are related to the symmetry operation 1 – x, y, ½ – z.

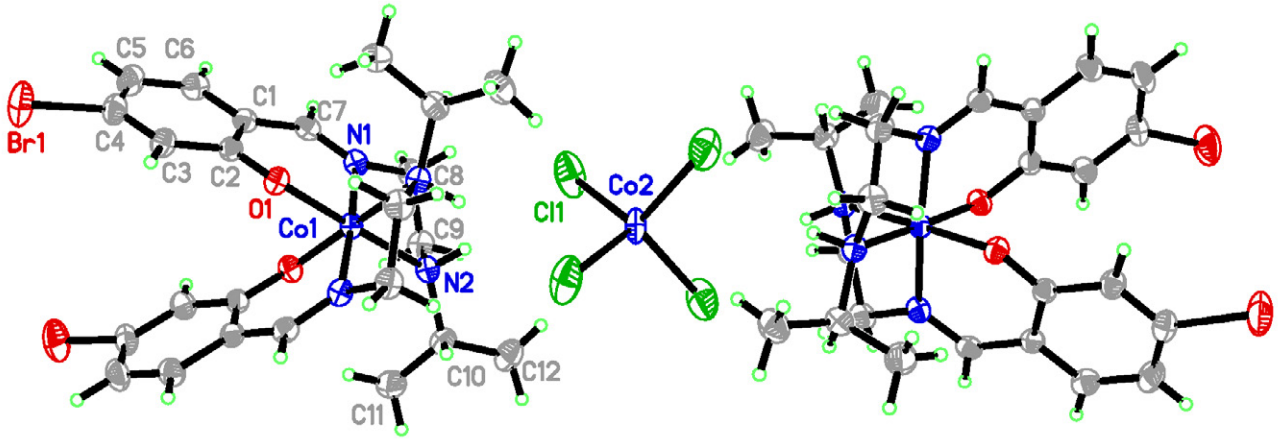


Fig. 2. Molecular structure of complex **2**. Unlabeled atoms are related to the symmetry operations 5/4 – x, y, 5/4 – z and 5/4 – x, 5/4 – y, z.

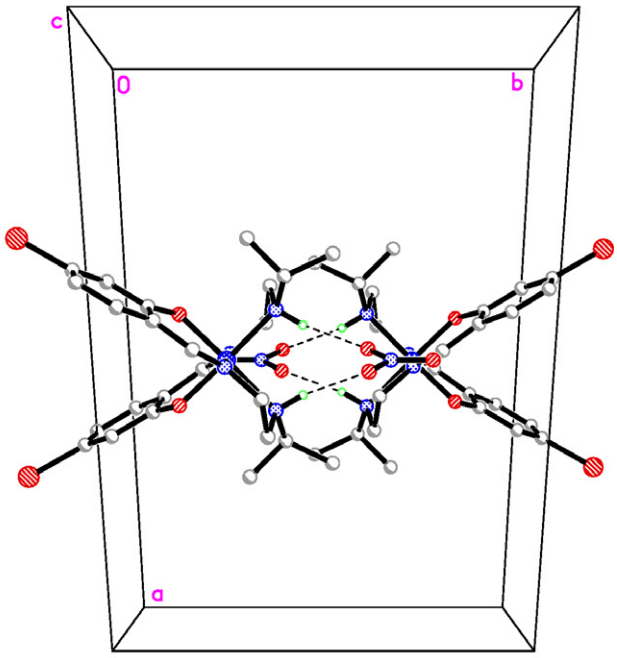


Fig. 3. Molecular packing structure of complex **1**, viewed along the *c* axis. Hydrogen bonds are shown as dashed lines.

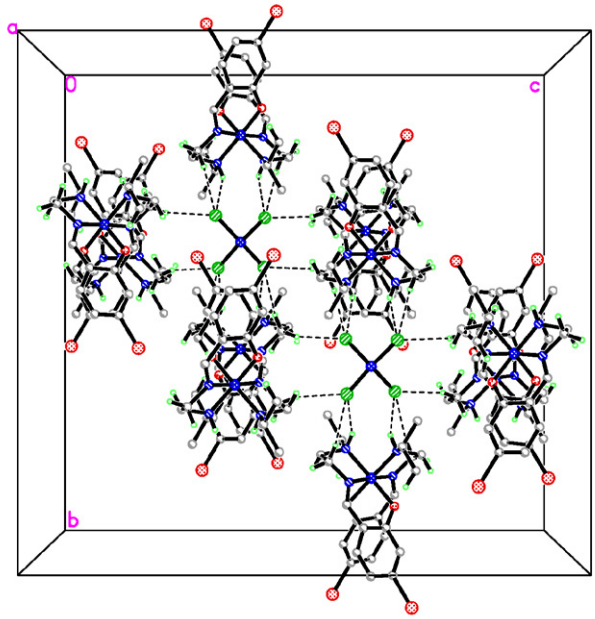


Fig. 4. Molecular packing structure of complex **2**, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

tions among the molecules (Cg(4)···Cg(4)^{c} 4.461(5) Å, symmetry code: c) ¼ – *x*, *y*, ¼ – *z*; Cg(4) is the centroid of C(1)–C(2)–C(3)–C(4)–C(5)–C(6)).

3. 4. Catalytic Property

The catalytic oxidation of cyclooctene with the two cobalt complexes as catalysts produced three products 9-oxa-bicyclo[6.1.0]nonane, cyclooct-2-enol and cyclooct-2-enone. Reaction conditions such as solvent, reaction time, catalyst, as well as oxidant to substrate ratio were optimized. Each catalyst (10 μmol) was dissolved in methanol (10 mL). Then, cyclooctene (15 mmol) and TBHP (30 mmol) were added. The mixture was stirred at reflux for 36 h. The reaction was monitored at 60 min intervals by gas chromatography. As a result, the conversion increased up to 20 h and reached the limit. Thus, 20 h was chosen as the optimized reaction time. Both complexes have similar properties and complex **1** was chosen to investigate the effect of other variables. The solvent type, catalyst load, and the oxidant to substrate ratio were shown as Fig. 5. Methanol is the best solvent for this reaction. The conversion of both complexes as catalysts is over than 75%, and epoxide selectivity is over 56% (Table 4). The catalytic performance of the present complexes is comparable with those reported in literature.[20]

Table 4. The catalytic results for the two cobalt complexes.

Complex	Conversion (%)	Selectivity (%)		TON
		Epoxide	Other products	
1	78	58	42	1167
2	75	56	44	1128

Table 3. Hydrogen bond distances (Å) and bond angles (°) for the complexes

D–H···*A*	*d*(*D*–H)	*d*(H···*A*)	*d*(*D*···*A*)	Angle (*D*–H···*A*)
1				
N(2)–H(2)···O(2)$^{\#1}$	0.91	2.59	3.375(3)	146(5)
N(2)–H(2)···O(2)$^{\#2}$	0.91	2.18	3.059(3)	161(5)
2				
N(2)–H(2)···Cl(1)$^{\#3}$	0.91	2.68	3.501(4)	151(6)
C(8)–H(8A)···Cl(1)$^{\#3}$	0.97	2.66	3.478(4)	142(6)
C(8)–H(8B)···Cl(1)$^{\#4}$	0.97	2.66	3.585(4)	159(6)
C(11)–H(11A)···O(1)$^{\#5}$	0.96	2.46	3.151(4)	129(6)

Symmetry codes: #1: –*x*, –*y*, 1 – *z*; #2: *x*, –*y*, –½ + *z*; #3: –½ + *x*, ½ + *y*, *z*; #4: ½ – *x*, ½ – *y*, –*z*; #5: ¼ – *x*, *y*, ¼ – *z*.

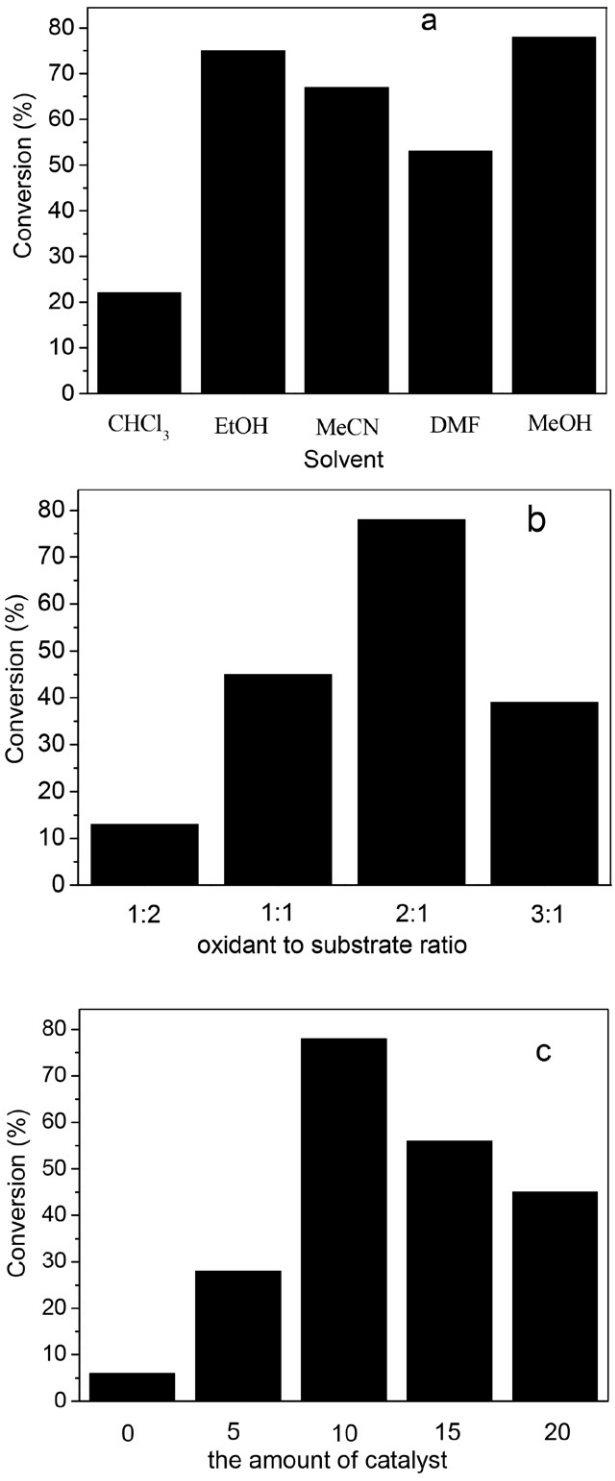


Fig. 5. The influence of solvent type (a), the oxidant to substrate ratio (b) and the amount of catalyst (c).

4. Conclusion

Two new cobalt complexes have been prepared and characterized. Structures of the complexes were characterized by spectroscopic methods, and confirmed by single crystal X-ray determination. One complex is a nitrate salt of a cobalt(III) complex, and the other one is a cobalt(II) tetrachloride salt of a cobalt(III) complex. The Schiff base ligand coordinates to the cobalt atoms through phenolate oxygen, imino nitrogen and amino nitrogen atoms. Both complexes are good catalysts for the oxidation of cyclooctene with TBHP as oxidant and methanol as solvent. The catalytic activity and epoxide selectivity were in high level.

Supplementary Material

CCDC reference numbers 2061676 (**1**) and 2061677 (**2**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk, or from Cambridge Crystallographic Data Center, 12, Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336 033; e-mail: deposit@ccdc.cam.ac.uk.

Acknowledgments

We gratefully acknowledge the financial support by the National Natural Science Foundation of China (22361001), the Natural Science Foundation of Inner Mongolia Autonomous Region (2023LHMS05032), and the Natural Science Foundation of Inner Mongolia Autonomous Region (2025MS02028).

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Povzetek

Pripravili smo dva nova kobaltova(III) kompleksa, $[CoL_2]NO_3$ (**1**) and $[CoL_2]_2[CoCl_4]$ (**2**), kjer je L 5-bromo-2-(((2-izopropilamino)etil)imino)metil)fenolat, ter ju okarakterizirali s fizikalno-kemijskimi metodami in monokristalno rentgensko analizo. Rentgenska analiza je pokazala, da so atomi Co v obeh kompleksih oktaedrično koordinirani, razen v enoti $[CoCl_4]$, kjer so tetraedrično koordinirani. Kristalne strukture kompleksov stabilizirajo vodikove vezi in π···π interakcije. Katalitične lastnosti obeh kobaltovih kompleksov smo preučevali na epoksidaciji ciklooktena s *t*-butilhidroperoksidom (TBHP) kot oksidantom. Oba kompleksa kažeta dobro katalitično aktivnost in visoko selektivnost.