

邻香兰素缩对甲苯胺 Schiff 碱过渡金属配合物的合成、 表征、晶体结构及抗菌活性研究

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摘要: 合成了邻香兰素缩对甲苯胺 Schiff 碱 HL(C₁₅H₁₅NO₂) 与锰(II), 锌(II)的配合物 [Mn(HL)₃(CH₃OH)](ClO₄)₂ (**1**) 和 [Zn(HL)₂(H₂O)₂](ClO₄)₂ (**2**)。用元素分析、红外光谱、紫外-可见光谱、¹H NMR 核磁共振、摩尔电导等手段进行表征; 用单晶 X-射线衍射方法测定了配合物的晶体结构, 配合物的中心金属离子与 Schiff 碱配体中的酚羟基氧及甲氧基氧发生配位, 锰(II)的配位数为 7, 而锌(II)的配位数为 6。测定了配体和配合物对大肠杆菌、金黄色葡萄球菌、枯草杆菌的抗菌活性。

关键词: 过渡金属配合物; 邻香兰素; 对甲苯胺; Schiff 碱; 晶体结构; 抗菌活性

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Synthesis, Characterization, Crystal Structures and Antibacterial Activities of Transition Metal(II) Complexes with a Schiff Base Derived from *o*-vanillin and *p*-toluidine

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Abstract: Two complexes [Mn(HL)₃(CH₃OH)](ClO₄)₂ (**1**), [Zn(HL)₂(H₂O)₂](ClO₄)₂ (**2**), where HL is a Schiff base ligand 2-[(4-methylphenylimino)methyl]-6-methoxyphenol (C₁₅H₁₅NO₂) derived from condensation of *o*-vanillin (2-hydroxy-3-methoxybenzaldehyde) with *p*-toluidine. They were characterized by elemental analysis, molar conductance, FTIR, UV-Vis, ¹H NMR spectra. The structures of complexes were determined by X-ray diffraction single-crystal structure analyses. The Schiff base ligand and its complexes have been tested *in vitro* to evaluate their antibacterial activity against bacteria, viz., *Escherichia Coli*, *Staphylococcus Aureus* and *Bacillus Subtilis*. It has been found that the Zn(II) complex has higher activity than the corresponding free Schiff base ligand and Mn(II) complex against the same bacteria. CCDC: 692600, **1**; 692601, **2**.

Key words: transition metal(II) complex; *o*-vanillin; *p*-toluidine; Schiff base; crystal structure; antibacterial activities

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The condensation of primary amines with carbonyl compounds yields Schiff bases^[1,2]. Schiff base ligands which usually contain O, N donor atoms play an important role in coordination chemistry since the late 19th century. Metal complexes with these ligands are becoming increasingly important as biochemical, analytical and antimicrobial reagents, design of molecular ferromagnets, materials chemistry and so on^[3-11]. Metal complexes with Schiff's base ligands resulting from condensation of salicylaldehyde and its derivatives with a primary amine have been studied for many years. Among these studies a relatively small number has involved Schiff's base in which the primary amine is aniline and its *o*-, *m*-, *p*-substituted derivatives^[12,13]. They behave as bidentate ligands and form complexes of the type M(HL)₂ with divalent first row metal ions^[14-17]. A large number of reports are available on the chemistry and the biocidal activities of transition metal complexes containing O, N donor atoms^[18-22]. Some literature survey reveal that Schiff base derived from *o*-vanillin and *p*-toluidine has been prepared, its metal complexes and biological activity have scarcely been investigated^[23-25]. In view of this, we describe the synthesis, characterization, single crystal structure and antibacterial properties of Mn(II) and Zn(II) complexes with a Schiff base in the neutral form 2-[(4-methylphenylimino)methyl]-6-methoxyphenol.

1 Experimental

1.1 Materials and general methods

o-vanillin, *p*-toluidine, and other chemical reagents were obtained from commercial sources and used without further purification. M(ClO₄)₂·6H₂O were made by the reaction of MO and HClO₄. The metal contents were determined by EDTA complexometric titration after decomposition a known amount of the complexes with concentrated nitric acid. Elemental analyses for C, H and N were carried out on an Elementar Vario EL III elemental analyzer. IR spectra on KBr pellets were recorded on a Nicolet NEXUS 670 FTIR spectrophotometer in the range of 4 000~400 cm⁻¹. Molar conductivity of the complexes were measured with a Shanghai DDS-11A conductivity meter in DMF (1.0×10⁻³ mol ·

dm⁻³). The ¹H NMR spectra were recorded at 400 MHz on a Bruker-400 spectrometer using d₆-DMSO as solvent, chemical shifts are expressed as δ (ppm) with respect to tetramethylsilane as an external reference. UV-Vis spectra of Schiff base ligand and the complexes were measured with a Shimadzu UV-2501PC spectrophotometer in methanol (1.0×10⁻⁴ mol · dm⁻³) at the range of 200~700 nm.

1.2 Synthesis of the Schiff base ligand

Schiff base ligand (HL) was prepared by the direct reaction of equimolar quantities of the *o*-vanillin (3.46 g, 0.03 mol) and *p*-toluidine (3.21 g, 0.03 mol) in 50 mL absolute ethanol. The reaction mixture was stirred for 30 min at room temperature, then the orange solid ligand were isolated by filtration, washed with ethanol and dried in air, Yield 5.20 g (86%), m.p. 100 °C. It was recrystallized in ethanol before used and further characterized by IR, ¹H NMR, UV-Vis spectroscopy as reported in the literature^[25]. IR (KBr, cm⁻¹); 3 450(w), 1 614 (C=N, s), 1 258 (C-O, s). ¹H NMR (400 MHz, DMSO-d₆, ppm); δ 13.38 (s, 1H, -OH), 8.94 (s, 1H, -CH=N-), 7.32 (dd, 2H, *J*=8.0 Hz, MePh-H), 7.26 (dd, 2H, *J*=8.0 Hz, MePh-H), 7.22~6.87 (m, 3H, Ph-H), 3.81 (s, 3H, -OCH₃), 2.33 (s, 3H, -CH₃). UV-Vis (λ / nm, ε / m² · mol⁻¹); 317.5 (335.0), 277.5 (248.7), 227.0 (498.1). Anal. Calcd. for C₁₅H₁₅NO₂ (241.29) (%): C 74.66, H 6.27, N 5.81; found (%) C 74.62, H 6.31, N 5.77.

1.3 Synthesis of solid complexes

A solution of Mn(ClO₄)₂·6H₂O (0.37 g, 1 mmol) in 10 mL absolute alcohol was added dropwise with stirring to a ethanol solution of Schiff base ligand (2.0 mmol, 0.48 g) for 4 h, then the clear red mixture solution was resulted. The red single crystals [Mn(HL)₃(CH₃OH)](ClO₄)₂ (**1**) were precipitated after 5 days at room temperature. The precipitate was separated by filtration, washed with absolute alcohol and finally air-dried. Yield, 0.34 g (40%). IR (KBr, cm⁻¹); 3 416(m), 1 638(C=N, s), 1 239(C-O, s), 1 124(s), 1 107(s), 622(s), 497(Mn-O, w). UV-Vis (λ / nm, ε / (m² · mol⁻¹)): 317.5 (853.2), 277.5 (634.3), 228.5 (1197.5). Molar conductivity (Λ_M / (S · cm² · mol⁻¹)): 117.0. Anal. Calcd. for C₄₆H₄₉Cl₂N₃O₁₅Mn (**1**) (1 009.75): C 45.92; H 4.89; N 4.16; Mn 5.44; found C 45.83, H 4.83, N 4.11, Mn 5.41. Pale

yellow single crystals (0.35 g, 41%) of $[\text{ZnL}_2(\text{H}_2\text{O})_2](\text{ClO}_4)_2$ (**2**) were obtained suitable for X-ray diffraction after 8 days by slow evaporation at room temperature. IR (KBr, cm^{-1}): 3 417 (m), 1 641 (C=N, s), 1 236 (C-O, s), 1 120 (s), 1 106 (s), 622 (s), 502 (Zn-O, w). ^1H NMR (400 MHz, DMSO- d_6 , ppm): δ 13.40 (s, 1H, -OH), 8.95 (s, 1H, -CH=N-), 7.34 (dd, 2H, $J=8.0$ Hz, MePh-H), 7.27 (dd, 2H, $J=8.0$ Hz, MePh-H), 7.24~6.90 (m, 3H, Ph-H), 3.82 (s, 3H, -OCH₃), 2.34 (s, 3H, -CH₃). UV-Vis (λ / nm, ε / $\text{m}^2 \cdot \text{mol}^{-1}$): 316.0 (668.0), 285.0 (492.8), 231.5 (937.3). Molar conductivity (Λ_m , $\text{S} \cdot \text{cm}^2 \cdot \text{mol}^{-1}$): 119.0. Anal. Calcd. for $\text{C}_{30}\text{H}_{34}\text{Cl}_2\text{N}_2\text{O}_{14}\text{Zn}$ (**2**) (782.86): C 46.02; H 4.38; N 3.58; Zn 8.35; found C 46.31; H 4.45; N 3.52; Zn 8.21.

1.4 Crystal structure determination

Single crystals with approximate dimensions 0.18

$\text{mm} \times 0.16 \text{ mm} \times 0.10 \text{ mm}$ for **1** and $0.30 \text{ mm} \times 0.28 \text{ mm} \times 0.10 \text{ mm}$ for **2** were selected and coated with vaseline. Intensity data for the complexes **1** and **2** were measured with a Rigaku R-Axis RAPID diffractometer with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.071\ 073 \text{ nm}$) at 296 K. Empirical absorption corrections were applied by use of the ABSCOR program. The structures were solved by direct methods and all calculations were performed with the aid of the SHELX-97 PC program^[26]. The structures were refined by full-matrix, least-squares minimization of $\sum (F_o - F_c)^2$ with anisotropic thermal parameters for all atoms except H atoms. The crystal data of the complexes **1** and **2** are summarized in Table 1, and the selected bond lengths and angles are in Table 2.

CCDC: 692600, **1**; 692601, **2**.

Table 1 Crystal data and structure refinement for **1** and **2**

	1	2
Empirical formula	$\text{C}_{46}\text{H}_{49}\text{MnN}_3\text{O}_{15}\text{Cl}_2$	$\text{C}_{30}\text{H}_{34}\text{ZnN}_2\text{O}_{14}\text{Cl}_2$
Formula weight	1 009.75	782.86
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/c$	$P\bar{1}$
Crystal color	Dark red	Yellow
a / nm	1.892 8(4)	0.741 59(15)
b / nm	1.223 1(2)	1.113 1(2)
c / nm	2.062 7(4)	1.127 2(2)
α / ($^\circ$)		80.31(3)
β / ($^\circ$)	95.53(3)	72.02(3)
γ / ($^\circ$)		70.83(3)
V / nm^3	4.753 1(16)	0.833 6(3)
Z	4	1
D_c / ($\text{g} \cdot \text{cm}^{-3}$)	0.984	1.559
μ / mm^{-1}	0.430	0.969
$F(000)$	2096	404
Temperature / K	293(2)	296(2)
λ (Mo $K\alpha$) / nm	0.071 073	0.071 073
Reflections collected / unique (R_{int})	40 171 / 10 608 (0.0493)	5 306 / 3 591 (0.0167)
Obsd. reflns. [$I > 2\sigma(I)$]	5 297	2 851
Parameters	616	231
S on F^2	1.045	1.031
R_1 [$I > 2\sigma(I)$]	0.064 6	0.048 6
wR_2 [$I > 2\sigma(I)$]	0.177 1	0.129 1
R_1 (all data)	0.177 1	0.064 2
wR_2 (all data)	0.2128	0.1400
Max, min $\Delta\rho$ / ($\text{e} \cdot \text{nm}^{-3}$)	401, -346	523, -474

Table 2 Selected bond lengths (nm) and angles (°) for **1** and **2**

1					
Mn(1)-O(1)	0.205 9(2)	Mn(1)-O(6)	0.238 3(3)	Mn(1)-O(3)	0.208 1(3)
Mn(1)-O(15)	0.224 4(3)	Mn(1)-O(5)	0.207 7(2)		
O(1)-Mn(1)-O(5)	156.07(11)	O(3)-Mn(1)-O(15)	78.35(12)	O(1)-Mn(1)-O(3)	106.40(11)
O(1)-Mn(1)-O(6)	88.79(10)	O(5)-Mn(1)-O(3)	97.32(11)	O(5)-Mn(1)-O(6)	71.25(9)
O(1)-Mn(1)-O(15)	100.12(13)	O(3)-Mn(1)-O(6)	137.25(11)	O(5)-Mn(1)-O(15)	87.52(12)
O(15)-Mn(1)-O(6)	139.14(12)				
2					
Zn(1)-O(1)#1	0.200 89(19)	Zn(1)-O(1W)	0.210 5(3)	Zn(1)-O(1)	0.200 89(19)
Zn(1)-O(2)#1	0.215 3(2)	Zn(1)-O(1W)#1	0.210 5(3)	Zn(1)-O(2)	0.215 3(2)
O(1)#1-Zn(1)-O(1)	180.00(12)	O(1W)#1-Zn(1)-O(2)#1	88.05(11)	O(1)#1-Zn(1)-O(1W)#1	89.92(11)
O(1W)-Zn(1)-O(2)#1	91.95(11)	O(1)-Zn(1)-O(1W)#1	90.08(11)	O(1)#1-Zn(1)-O(2)	101.18(8)
O(1)#1-Zn(1)-O(1W)	90.08(11)	O(1)-Zn(1)-O(2)	78.82(8)	O(1)-Zn(1)-O(1W)	89.92(11)
O(1W)#1-Zn(1)-O(2)	91.95(11)	O(1W)#1-Zn(1)-O(1W)	180.00(19)	O(1W)-Zn(1)-O(2)	88.05(11)
O(1)#1-Zn(1)-O(2)#1	78.82(8)	O(2)#1-Zn(1)-O(2)	180.00(11)	O(1)-Zn(1)-O(2)#1	101.18(8)

Symmetry codes: #1: $-x, -y, -z$.

2 Results and discussion

Elemental contents of C, H, N, M both theoretically calculated values and actual values are in accordance for their formula of the complexes, and it shows that the Schiff base ligand is neutral. This can be explained by the absences of any deprotonating agent during the synthesis. Complexes in which a Schiff base coordinates as a neutral ligand are still rare. Recent studies^[27,28] refer to complexes of M(II) ion in which the Schiff base coordinates in this unusual structural characteristic via the phenolic hydroxy oxygen atom, the nitrogen atom remaining uncoordinated. Their molar conductance in DMF solvent suggested that the complexes **1**~**2** were 2:1 electrolytic nature^[29].

2.1 IR spectra

The broad absorption band at 3450 cm^{-1} attributed to the hydroxy group in the spectra of the free ligand are enhanced and appear at lower frequency in the corresponding complexes, viz., 3416 cm^{-1} (**1**), 3417 cm^{-1} (**2**), showing coordination of oxygen atom of the phenolic hydroxy with the central M(II) ion. The shift of the C-O stretching vibration of the phenolic part of o-vanillin from 1258 cm^{-1} to 1239 cm^{-1} (**1**), 1236 cm^{-1} (**2**) also supports the coordination of oxygen atoms. However, a

strong band in the free Schiff base ligand occurring at 1617 cm^{-1} attributed to C=N stretching is found shifted to higher frequency, viz., 1638 cm^{-1} (**1**), 1641 cm^{-1} (**2**), but the nitrogen atom of azomethine was regarded as no complexation with M(II)^[27,28,30]. And also, a new band at 497 cm^{-1} (**1**), 502 cm^{-1} (**2**), attributed to M-O stretching vibration, whereas it was none in ligand. In the spectra of complexes, perchlorate anions show two sharp bands at $1120\sim 1124\text{ cm}^{-1}$, $1106\sim 1107\text{ cm}^{-1}$ and a medium stretching band at 622 cm^{-1} which are attributed to the non-coordinating perchlorate anions^[30]. A band assignable to the stretching of O-H bond is found to be broadened within the frequency range of $3050\sim 2700\text{ cm}^{-1}$. This observation implies that the H atom from the OH group in free and complexation Schiff base ligand has a tendency to migrate to azomethine N atom via the O-H \cdots N intramolecular hydrogen bonding as that reported in the paper^[25].

2.2 ^1H NMR spectra

^1H NMR of complex **2**, for example, shows the following important proton signals: 8.95 ppm assigned to the imine CH=N proton; 3.82 ppm assigned to the methoxy -OCH₃ protons and 2.34 ppm assigned to the methyl -CH₃ protons. The signal in free ligand appeared as a singlet at $\delta=13.38$ ppm can be ascribed to OH, as

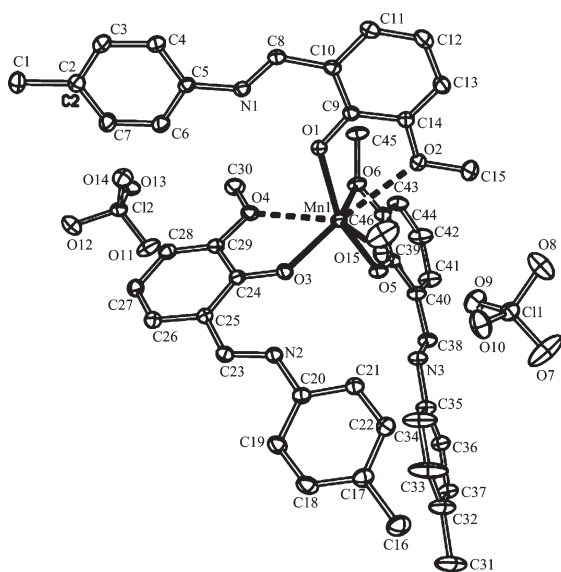
that observed in the literature^[25]. Which was not almost changed in the corresponding complex ($\delta=13.40$), indicating that the O—H...N intramolecular hydrogen bonding due to the H atom from the O—H group to azomethine N atom was still present in complexes.

2.3 UV-Vis spectra

Three absorption peaks of the free Schiff base ligand appeared at 317.5 nm, 277.5 nm and 227.0 nm in the range of 200~400 nm. The band of 317.5 nm may be assigned to $n-\pi^*$ transition of conjugation between lone-pair electron of p orbital of N atom in C=N group and big π bond of benzene ring. A peak at 277.5 nm is assigned to $\pi-\pi^*$ transition of conjugation system of Schiff base. The spectra of two complexes are very similar and almost the same as Schiff base ligand.

2.4 Crystal structures

[Mn(HL)₃(MeOH)](ClO₄)₂ (**1**): A local environment of manganese of complex **1** is shown in Fig.1. The asymmetry unit consists of one Mn atom, three HL ligands, one methanol molecule and two perchlorate ions. It is interesting to note that there is a weak interaction between Mn(II) and O2, O4 atoms with Mn—O distance of 0.255 91(33) and 0.263 18(28) nm. It is longer than that of the Mn—O bond in the complex [Mn(C₈H₇O₃)₂(H₂O)₂] (0.235 06 nm)^[31], but shorter than van der Waals radius in Mn—O (0.337 nm). Thus, the environment of the manganese(II) can be described as

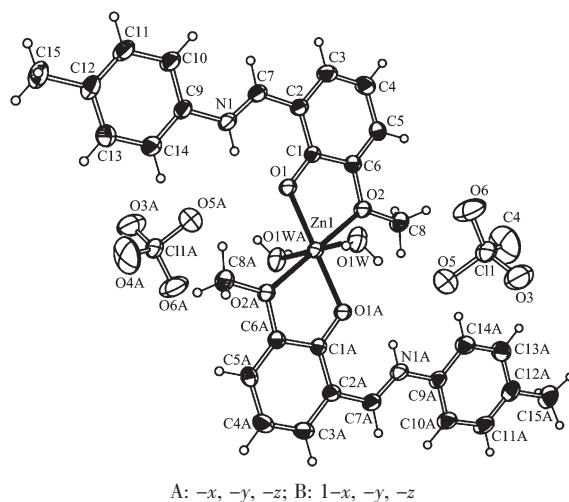


Displacement ellipsoids are drawn at the 10% probability level

Fig.1 Molecular structure of **1**

seven coordinate, with an approximately pentagonal bipyramidal O7 coordination sphere. The axial donors are O1 and O5 atoms, the pentagonal plane is occupied by the HL and methanol O atoms. The Mn—O distances vary from 0.205 9(2) to 0.263 18(28) nm. The phenyl rings (C2—C7 and C9—C14) are almost parallel to each other, with dihedral angles of only 0.572°, it is more smaller than that in the similar Schiff base ligand^[32-34]. The perchlorate anions are interspersed homogeneously among the cations.

[Zn(HL)₂(H₂O)₂](ClO₄)₂ (**2**): A local environment of zinc of complex **2** is shown in Fig.2. The asymmetry unit consists of half a zinc atom, one HL ligand, one water molecule and one perchlorate ion. In the [Zn(HL)₂(H₂O)₂] cation unit, the center Zn atom is six-coordinated by four O atoms of two HL ligands and two water O atoms, and displays slightly octahedral coordination geometry, with Zn—O distances in the range of 0.200 89(19)~0.215 3(2) nm. Four O atoms of two HL ligands determine the equatorial plane and Zn atom lies just this plane, and two water O atoms occupy the apical position. The Zn—O distances in this complex is comparable to those found in other Zn(II) complexes^[35]. The angle between two phenyl rings is 11.21°. The perchlorate anions are interspersed homogeneously among the cations.



Displacement ellipsoids are drawn at the 30% probability level

Fig.2 Molecular structure of **2**

2.5 Antibacterial activity

The *in vitro* antibacterial screening of the Schiff

base ligand and its complexes dissolved in DMF has been carried out against three bacteria, viz., *Escherichia Coli*, *Staphylococcus Aureus* and *Bacillus Subtilis*, with a method of filter paper^[36], using agar medium and the concentration of test solutions is 5 mg·mL⁻¹. Table 3 shows the zone of inhibition for the test solutions on three bacteria. It has been observed from the data that both the Schiff base ligand and its

complexes exhibit antibacterial activities against these three bacteria. It is noteworthy that the Zn(II) complex **2** has higher activity than Mn(II) complex **1** and the corresponding free Schiff base ligand against the same bacteria. Mn(II) complex has higher activity than ligand for *Bacillus Subtilis* but lower activity than ligand for *Escherichia Coli* and *Staphylococcus Aureus*.

Table 3 Inhibitory of compounds against bacteria growth

Compound	Diameter of inhibition zone / mm		
	<i>Escherichia Coli</i>	<i>Staphylococcus Aureus</i>	<i>Bacillus Subtilis</i>
HL	12.6	11.3	11.8
1	12.2	10.3	13.7
2	15.7	16.7	19.2

Note: Data in the table are the average values of three experiments, and the diameter of filter paper is 10 mm.

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规模最大的化学年会, 交流论文近三千篇 ——中国化学会第26届学术年会在天津举行

中国化学会第26届学术年会2008年7月13日至16日在天津南开大学举行。这是中国化学会历史上规模最大的一次学术活动。有包括31位两院院士在内的2800余名海内外化学界的专家学者到会。年会以“化学与和谐社会”为主题展开学术交流。此次会议由中国化学会主办、南开大学承办,天津市化学会、天津师范大学、天津理工大学协办。美国化学会、英国皇家化学会、德国化学会等国外学术团体应邀参加了盛会,与会外宾达40余人。

中国化学会理事长、中国科学院常务副院长、大会主席白春礼院士致开幕词,中国化学会副理事长、南开大学教授、大会执行主席程津培院士讲话;中国科协书记处书记、中国化学会常务理事冯长根,南开大学校长饶子和院士,美国化学会主席布鲁斯·伯斯坦(Bruce Bursten),英国皇家化学会首席执行官理查德·派克(Richard Pike)博士,德国化学会代表、《德国应用化学杂志》总编彼得·格里茨(Peter Göltz)分别致贺辞。国家自然科学基金委员会副主任、中国化学会秘书长姚建年院士主持了会议。开幕式在南开大学体育馆举行。

本届年会得到广大化学界同仁的热切关注,收到论文近3000篇,以大会特邀报告、分会邀请报告、专题报告与讨论、论文墙报展讲、专题学术论坛等形式展开交流。邀请了Kendall N. Houk、Astrid Gräslund、张希、朱道本、高松、宋礼成和龙军等7位著名中外学者做特邀报告;年会共设立绿色化学、环境化学、应用化学、纳米化学、无机与配位化学、有机固体材料、化学教育等20个分会和“化学与社会”论坛。

在“化学与社会”论坛上,闵恩泽、戴立信、王夔、赵玉芬、柴之芳5位院士和英国皇家化学会、BASF公司、保洁公司和公安、环保等部门的学者和企业家分别阐述了化学在建立和谐社会中的地位 and 作用。

开幕式上,颁发了2006、2007年度“中国化学会-约翰威立出版公司青年化学论文奖”,第四届“中国化学会-巴斯夫青年知识创新奖”,2007年度“中国化学会青年化学奖”,并颁发了首届“中国化学会-英国皇家化学会青年化学奖”和首届“中国化学会-中国石油化工股份有限公司化学贡献奖”。“中国化学会-中国石油化工股份有限公司化学贡献奖”是目前中国化学会的最高奖励,用于表彰在化学基础研究和应用研究中做出卓越贡献的化学家。朱道本院士和龙军教授获得此项殊荣,共计25人获得上述奖励。

年会组委会向参加年会的59名青年颁发了“优秀青年化学论文奖”,同时向11个分会的负责同志授予“优秀组织奖”。

年会期间还召开美国著名化学杂志《化学研究评论》(Accounts of Chemical Research)编委会、“中美合作化学研究生项目研讨会”;举办了“新技术、新产品与新仪器成果展”,近50家企业参加展出和交流。

本届年会于7月16日下午在南开大学体育馆胜利闭幕。会议得到化学、化工各界的18家高校、研究机构和企业的支持和赞助。