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Trigonal dodecahedral sodium coordination in a trinuclear copper(II)-sodium complex incorporating a salen-type compartmental Schiff base

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Abstract: A new trinuclear heterometallic complex, $[(CuL)Na(CuL)] \cdot ClO_4$ (1), has been prepared using a Schiff base, H₂L (where H₂L=*N*,*N*'-(1,2-phenylene)-bis(3-methoxysalicylideneimine) and characterized by elemental analysis, Fourier transform infra-red (FT-IR) spectroscopy, UV/Vis, magnetic, electrochemical, and single crystal X-ray diffraction methods. The structure analysis reveals that two metallo-ligand [(CuL)] units are connected to each other by a sodium ion resulting in the cationic unit [(CuL)Na(CuL)]⁺. Both the copper(II) ions display an almost square planar geometry while the sodium ion adopts a trigonal-dodeca-hedral coordination geometry. The spectroscopic and other physicochemical studies are in good agreement with the crystal structure of the complex.

Keywords: crystal structure; cyclic voltammetry; Schiff base; trigonal dodecahedral sodium coordination; tri-nuclear copper(II)-sodium.

1 Introduction

The coordination chemistry of the alkali metal ions received enormous interest over the past several decades aiming to develop molecular systems that can mimic naturally occurring molecules responsible for the selective transport of ions [1–3]. Besides several well-known classes of complexing agents such as crown ethers, cryptands etc. other simpler and easily affordable ligands are also used for this purpose and help to study the structure/ selectivity relationship of the alkali metal ions [4–6]. The salicylaldimine or acetylacetoneimine complexes of divalent metal ions serve the role of these complexing agents for their abilities to bridge other metal ions through their phenoxo oxygen atoms. Thus these classes of 'ligand complexes' play an important role for constructing varieties of homo-/heterometallic complexes including alkali metal ions, p- and d-block elements [7–9]. The role of compartmental dinucleating ligands can also be mentioned in this regard for their abilities to form homo-/ heterometallic complexes. These compartmental ligands may either by obtained by 2:1 condensation of varieties of substituted salicylaldehyde (3-methoxy/3-ethoxy/3carboxy) and a diamine or by 2:1:1 condensation of a diformyl compound with two different diamines (such as 4-methyl-2,6-diformylphenol, 1,3-propanediamine, and ethylenediamine) leading to two dissimilar or similar compartments with respect to the donor sets and capable of accommodating two metal ions of similar/different types generating varieties of homo-/heterometallic dinuclear compounds [10]. Moreover, both 'ligand complexes' and dinuclear compounds derived from bicompartmental Schiff bases are further considered as small building blocks which in combination with other metal ions or even in the presence/absence of other secondary bridging ligands can undergo self-assembly to produce oligonuclear and polymeric systems, which is one of the most important developments in the field of supramolecular chemistry [7, 10, 11]. Inclusion of small molecules/ions by these 'ligand complexes' or complexes of compartmental Schiff bases plays an important role in host-guest chemistry [7, 12]. Due to the presence of highly unsaturated and delocalized ligand moieties the electron enrichment in these complexes is possible because of π multiple metalligand interactions which made these complexes as electron reservoirs for molecular activation processes. These complexes also find application as carriers for polar organometallics, and ferroelectricity [13].

Compartmental salen-type Schiff bases as multi-nucleating agents based on different flexible

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aliphatic diamines were well explored while their alicyclic and aromatic diamine analogs are few in number [10, 14–18]. In this context, the role of the compartmental Schiff base, H₂L, *N*,*N'*-(1,2-phenylene)-bis(3methoxysalicylideneimine) (Scheme 1), obtained by the condensation of 3-methoxysalicylaldehyde and *o*-phenylenediamine, as multi-nucleating agents was explored in a number of heteronuclear 3d/ns [3, 10, 19], and 3d/nf [14, 20–22], 3d/np [23, 24] metal compounds, where the 3d metal ion gets accommodated in the inner N₂O₂ core while the outer O₂O₂ compartment suits for the *ns/np/nd/nf* metal ions. The compounds isolated can be formulated as LMM'X or LML*n*X, where L is the ligand, M is the 3d metal ion [mainly, Cu(II)/Zn(II)], Ln is the 4f/5f metal ion, and X is the anionic species [25, 26].

However, trinuclear cationic M_2M' compounds derived from this compartmental ligand (H_2L) with ClO_4^- as a counter ion are extremely rare [27]. Herein, we report the synthesis, spectral and structural characterization of a new trinuclear compound [(CuL)Na(CuL)]ClO₄ (1). The structural characterization identifies the sodium ion in a trigonal dodecahedral coordination geometry which is believed to be rare in this class of compounds. Room temperature magnetic and electrochemical properties of the complex we also investigated.

2 Results and discussion

2.1 Fourier transform infrared (FT-IR) spectrum

The FT-IR spectrum of the complex is in accordance with the structure of the compound. The spectrum reveals that

the azomethine (-CH=N-) stretching vibration of the coordinated ligand is observed at 1603 cm⁻¹ in comparison to 1615 cm⁻¹ observed for the free Schiff base [28]. Phenolic -OH stretching vibration of the Schiff base observed as a broad band centred at 3349 cm⁻¹ is absent in the spectrum of the complex indicating deprotonation of this group during complexation, further confirmed by the observed lowering of the phenolic C–O stretching bands in comparison to that of the ligand [28]. The phenolic C–O stretching bands for the complex were observed at 1226 and 1145 cm $^{-1}$ in comparison to 1298 and 1236 cm⁻¹ observed for the ligand [29, 30]. The band at 3055 cm⁻¹ may be assigned to alkyl C–H bond stretching of the methoxy group [12]. Coordination of nitrogen atoms to the copper(II) center $(\nu_{C_{U-N}})$ is also evident by the appearance of weak bands at 484 and 425 cm⁻¹ for **1**. A sharp and strong peak at 1087 cm⁻¹ in the spectra of **1** may be assigned to perchlorate stretching vibration and this single peak without splitting is evident for the presence of a non-coordinating perchlorate anion [31].

2.2 UV/Vis spectrum

The electronic spectrum of **1** was recorded in acetonitrile at room temperature. The spectrum shows a very broad and low intensity absorption band at 562 nm corresponding to the $d \rightarrow d$ transitions of the square planar coordination of the copper(II) ion [3]. The complex also shows a strong and intense band at 366 nm which is believed to be due to ligand-to-metal charge transfer transitions (LMCT). Two very strong and intense bands in the higher energy region (272 and 222 nm) associated with $\pi \rightarrow \pi^*$ transitions of the coordinated ligand are also observed [3, 11, 32, 33].



Scheme 1: Synthetic scheme of Schiff base (H₂L) and coordination modes of L²⁻.

2.3 Crystal structure of $[(CuL)Na(CuL)] \cdot ClO_4$ (1)

An ORTEP view of the molecular structure of 1 is shown in Fig. 1 and the relevant bonding parameters are summarized in Table 1. The crystal structure reveals that the complex consists of a discrete trinuclear cationic unit [(CuL)Na(CuL)]+ and a disordered perchlorate ion as counter anion. In the cationic unit two CuL moieties are held together by a sodium ion. In the complex, the deprotonated Schiff base (L2-) simultaneously holds the copper(II) ion in its inner core using the imine nitrogen and phenolato oxygen atoms while it holds the sodium ion in its outer core through the same phenolato oxygen atoms involved in the coordination of the copper metals and the methoxo oxygen atoms. Thus each copper(II) center is tetra-coordinated by two imine nitrogen (N1 and N2 for Cu1; N3 and N4 for Cu2) and two phenolato oxygen atoms (O2 and O3 for Cu1; O6 and O7 for Cu2) and adopts an approximately square-planar geometry as evident from the bonding parameters (Table 1). The Cu1 and Cu2 atoms protrude by 0.636 (6) and 0.0628(5) Å from the respective least-squares coordination planes. The N₂O₂ donor sets

are slightly but significantly tetrahedrally distorted, the deviation from the planarity ranging from -0.051(7) to 0.042(4) Å and from -0.72(4) to 0.81(4) Å for N1/N2/O2/O3 and N3/N4/O6/O7, respectively.

The mean Cu–O (1.885(3) and 1.886(5) Å for Cu1 and Cu2) and Cu–N (1.921(8) and 1.928(5) Å for Cu1 and Cu2) bond lengths are not significantly different and in good agreement with values reported in the literature [11]. Moreover, Cu–O distances are significantly longer with respect to those observed in similar reported mononuclear complexes, possibly as a consequence of the bridging role of the phenolato oxygen atoms [2]. The transiod and cisoid angles deviate from their ideal values of 180 and 90° (Table 1) and the mean *trans* basal angles are the same within experimental errors [176(2) and 176(4)° for Cu1 and Cu2], indicating that both copper(II) atoms have a very similar distorted square planar coordination geometry. On the other hand, the sodium center is connected to two CuL moieties through the coordination of four phenolato oxygen (02, 03, 06, and 07) and four methoxy oxygen (01, 04, 05, and 08) atoms. Thus both copper(II) and sodium ions are connected directly through double μ -phenolato



Fig. 1: The molecular structure of 1 with displacement ellipsoids drawn at the 40% probability level. Only the major component of the disordered perchlorate anion is shown.

Cu1-02	1.885(3)	Na1-06	2.499(3)
Cu1-03	1.885(3)	Na1-02	2.503(3)
Cu1-N1	1.912(4)	Na1-07	2.518(3)
Cu1-N2	1.929(4)	Na1-03	2.531(3
Cu2-06	1.880(3)	Na1-05	2.604(3)
Cu2-07	1.890(3)	Na1-01	2.608(3)
Cu2-N3	1.923(4)	Na1-08	2.627(3)
Cu2-N4	1.932(3)	Na1-04	2.641(3)
02-Cu1-03	84.91(12)	07–Na1–05	121.35(11)
02-Cu1-N1	95.82(17)	03-Na1-05	89.04(10)
03-Cu1-N1	173.91(14)	06-Na1-01	86.77(11)
02-Cu1-N2	177.99(14)	02-Na1-01	60.74(10)
03-Cu1-N2	94.43(16)	07-Na1-01	80.11(10)
N1-Cu1-N2	84.63(19)	03-Na1-01	121.24(11)
06-Cu2-07	84.71(12)	05-Na1-01	99.21(11)
06-Cu2-N3	95.44(15)	06-Na1-08	120.75(11)
07-Cu2-N3	179.83(16)	02-Na1-08	78.94(10)
06-Cu2-N4	172.71(13)	07–Na1–08	60.00(9)
07-Cu2-N4	95.36(14)	03-Na1-08	87.52(10)
N3-Cu2-N4	84.48(17)	05–Na1–08	174.38(12)
06-Na1-02	142.05(12)	01-Na1-08	86.38(11)
06–Na1–07	60.84(10)	06-Na1-04	88.72(10)
02–Na1–07	124.73(11)	02-Na1-04	120.49(11)
06-Na1-03	142.80(11)	07–Na1–O4	104.15(10)
02-Na1-03	60.73(10)	03-Na1-04	59.91(9)
07–Na1–O3	141.21(12)	05–Na1–O4	72.06(10)
06–Na1–05	60.57(10)	01-Na1-04	171.27(12)
02–Na1–05	103.20(11)	08-Na1-04	102.35(11)

Table 1: Selected bond lengths (Å) and angles (deg) for complex 1.

Table 2: Hydrogen bonding parameters (Å, deg) for 1.ª

D–H…A	D-H	Н…А	DA	∠ D–H…A
C9–H9…O10 ^{#1}	0.93	2.53	3.207(8)	130
C22-H22A…O11 ^{#2}	0.96	2.51	3.457(8)	170
C27-H27…O12 ^{#3}	0.93	2.57	3.417(10)	152
C29-H29012 ^{#3}	0.93	2.51	3.383(9)	157

^aEquivalent atoms generated by symmetry codes: $^{#1}1-x$, -y, 1-z; $^{#2}1-x$, 1/2+y, 1/2-z; $^{#3}-x$, 1/2+y, 1/2-z.

bridges as well as through the coordination of methoxo oxygen atoms of the Schiff base moieties. The O_4 donor sets of the Schiff bases are essentially planar (r.m.s. deviation of 0.006 and 0.002 Å for O1/O2/O3/O4 and O5/O6/O7/O8, respectively) and almost orthogonal to each other, forming a dihedral angle of 77.09(6)°.

The geometry around sodium is best described as trigonal dodecaherdral (Fig. 2) which is rarely occurring except for sodium coordinated with adequate crown ether ligands. To the best of our knowledge this is the third example of its kind of trinuclear complexes [10, 27] and believed to be first within the copper(II)-sodium polynuclear family bearing this compartmental ligand.



Fig. 2: Trigonal dodecahedral coordination geometry of the sodium ion in 1.

In the coordination environment, the Na–O (phenolato) distances [mean value 2.53(3) Å] are significantly shorter than the Na–O(methoxo) distances [mean value 2.620(9) Å] which is in good agreement with the compounds reported earlier [10]. The intra-molecular Cu1…Cu2 separation is 7.0594 (8) Å while the Cu1…Na and Cu2…Na separations are of 3.556 and 3.5491(1) Å, respectively.

The packing of the molecules reveals that the perchlorate anions and the trinuclear cationic units are connected through unclassical C–H···O interactions (Table 2) leading to a three-dimensional supramolecular structure.

2.4 Magnetic study

Room temperature magnetic susceptibility measurements for **1** gave an effective magnetic moment (μ_{eff}) of 1.74 BM. This behavior is very similar to the spin-only magnetic moment of an isolated copper(II) system (d⁹, $S = \frac{1}{2}$), supporting the presence of magnetically non-coupled square planar copper(II) ions in the complex.

2.5 Electrochemical study

A cyclic voltammogram of **1** (Fig. 3) was recorded at room temperature in acetonitrile within the potential range of



Fig. 3: Cyclic voltammogram of 1 (vs. SCE) recorded in acetonitrile at room temperature.

-1.5 to +1.5 V using tetrabutylammonium perchlorate as supporting electrolyte measured at scan rates of 50, 100 and 150 mV s⁻¹. With cathodic scan a reductive response observed at -0.61, -0.66, and -0.69 V (versus SCE) may be assigned to $Cu(II) \rightarrow Cu(I)$ reduction while with anodic scan an oxidative response assignable to $Cu(I) \rightarrow Cu(II)$ oxidation is observed at - 0.92, - 0.88, and - 0.87 V (versus SCE) at the same scan rates of 50, 100, and 150 mV s⁻¹, respectively [7]. The irreversible nature of the redox process is indicated by the peak-to-peak separations of 310, 220, and 180 mV, at their respective scan rates. It can be further observed that on increasing the scan rate, the cathodic peak positions shift to more negative values and the anodic peak positions are observed at more positive values while peak currents increase in both scans and I_n, is always more than I_{na}, also confirming the irreversibility of the redox process.

Moreover, the presence of only one redox couple suggests that both copper(II) ions present in the compound are in a similar chemical environment as revealed by the single crystal X-ray structure of the compound. However, other irreversible oxidative response, tentatively assigned for the oxidation of the coordinated Schiff base, are observed at +0.91, +0.98, and +1.04 V during the anodic scan for the respective scan rates.

3 Conclusion

Herein we have reported the synthesis and spectrocscopic characterization of a rare trinuclear copper(II)–sodium complex incorporating an *o*-phenylene based salen-type compartmental Schiff base which is capable to form trinuclear compounds. The structural characterization reveals that the sodium ion in the complex adopts a trigonal dodecahedral coordination geometry which is very rare in this class of compounds. The electrochemical properties reveal a behavior similar to those observed in mononuclear copper(II)-salen-type compounds.

4 Experimental section

4.1 Materials

Caution! Perchlorate salts of metal ions in presence of organic ligand are potentially explosive. Though we did not encounter any problem, the compound should be prepared in small amount and handled with utmost care.

All chemicals and solvents used in the synthesis were of analytical grade. Sodium perchlorate monohydrate was purchased from E. Merck (Mumbai, India). *O*-Vanillin, *o*-phenylenediamine, and copper(II) perchlorate hexahydrate were purchased from Aldrich Chemical Co. Inc. (Sigma Aldrich, St. Louis, MO, USA) and were used as received.

4.2 Physical techniques

Elemental analyzes (carbon, hydrogen and nitrogen) were carried out with a Perkin Elmer 2400 II elemental analyzer (Perkin Elmer, MA, USA). Copper(II) content in the complex was estimated quantitatively using a standard iodometric procedure. The FT-IR spectra (4000-400 cm⁻¹) were recorded on a Perkin Elmer Spectrum RX I FT-IR system (Perkin Elmer, MA, USA) with solid KBr pellets. UV/Vis spectra in solution were recorded at room temperature on a PerkinElmer Lambda 40 UV/Vis spectrophotometer (Perkin Elmer, SC, USA) using acetonitrile in 1 cm quartz cuvettes. Magnetic susceptibilities were measured with a model 155 PAR vibrating sample magnetometer (E Mc Grath Inc., Salem, USA) fitted with a Waker Scientific 175 FBAL magnet using Hg[Co(SCN),] as the standard. Necessary diamagnetic corrections for the ligand were performed using Pascal's table. Electrochemical measurements were performed using a PAR VersaStat-potentiostat/Galvanostat II electrochemical analysis system (Princeton Applied research, Oak Ridge, USA) under dry argon using conventional three-electrode configurations in acetonitrile with tetrabutylammonium perchlorate as the supporting electrolyte. A platinized platinum millielectrode and a saturated calomel electrode (SCE) were used as working and reference electrodes, respectively, along with a platinum counter electrode in cyclic voltammetry performed at a scan rate of $\nu = 50$, 100, and 150 mV s⁻¹.

4.3 Preparation of the ligand and complex

4.3.1 Schiff base ligand: N,N'-(1,2-phenylene)-bis(3methoxysalicylideneimine (H,L)

The Schiff base ligand, H_2L , was prepared by refluxing *o*-vanillin (1.52 g, 10 mmol) *o*-phenylenediamine (0.540 g, 5 mmol) in 50 mL methanol for 1 h. The reaction mixture was filtered. The filtrate kept at room temperature for slow evaporation of the solvent. Deep orange crystals of the Schiff base ligand H_2L were separated which is filtered and air dried. Yield: 1.64 g (87%)

4.3.2 [(CuL)Na(CuL)]·ClO₄ (1)

A methanolic solution (20 mL) of the Schiff base H_2L (0.790 g, 2 mmol) was added to a methanolic solution

(10 mL) of copper(II) perchlorate hexahydrate (0.741 g, 2 mmol) with slow stirring. A greenish brown precipitate immediately separated out which was filtered and air dried. The precipitate was then dissolved in 20 mL *iso*-propanol with slow stirring. To the resulting solution another *iso*-propanolic solution (10 mL) of sodium perchlorate monohydrate (0.141 g, 1 mmol) was added dropwise with constant stirring. The reaction mixture was again stirred for another hour and filtered. The filtrate was kept undisturbed at room temperature. Diffraction quality air stable deep brown single crystals of **1** were separated after 3 weeks. Yield: 0.635 g (72%) with respect to metal substrate.

4.3.3 Physical and spectroscopic data

For H₂L: – UV/Vis (CH₃CN): λ_{max} (lg ε_{max})=332 (3.58), 282 (4.72), and 211 nm (4.75). – IR (KBr disc): ν =3349 (phenolic –OH), 1615 (–CH=N–), 1298, and 1236 (phenolic C–O) cm⁻¹. – C₂₂H₂₀N₂O₄ (376.41): calcd. C 70.20, H 5.36, N 7.44; found C 70.14, H 5.30, N 7.38.

For 1: - UV/Vis (CH₃CN): λ_{max} (lg ε_{max})=562 (2.74), 366 (4.40), 272 (4.56), and 222 nm (4.59). - IR (KBr disc): ν =3427, 3055 (methoxy C–H), 1627, 1603 (–CH=N–), 1580, 1518, 1494, 1423, 1341, 1319, 1307, 1226, and 1145 (phenolic C–O), 1111, 1087 (ClO₄⁻), 950, 852, 782, 738, 724, 653, 621, 575, 509, 484 (Cu–N), 457, 425 (Cu–N) cm⁻¹. - C₄₄H₃₆Cl-Cu₂N₄NaO₁₂ (998.31): calcd. C 52.94, H 3.63, N 5.61, Cu 12.73; found C 52.87, H 3.58, N 5.58, Cu 12.65.

4.4 Crystal structure determination

A diffraction quality air stable block shaped brown crystal of 1 was mounted on a Bruker SMART APEX II CCD area detector diffractometer equipped with a fine-focus, sealed tube X-ray source with graphite monochromated MoK α radiation (λ = 0.71073 Å). The data were collected by φ and ω scan modes at 294(2) K. Data collection was performed with the Bruker APEX2 [34] (Bruker AXS Inc., Madison, WI, USA) software while cell refinement and data reduction were performed with the Bruker SAINT program [34] (Bruker AXS Inc., Madison, WI, USA). Multiscan absorption corrections were applied to the intensity values (T_{max} =0.900 and T_{min} =0.769) using SADABS [34] (Bruker AXS Inc., Madison, WI, USA). The structure of 1 was solved by Direct Methods using the program SHELXT [35] (Institut Anorg. Chemie, Göttingen, Germany) and refined with full-matrix least-squares based on F^2 using SHELXL-2014/7 [36, 37] (Institut Anorg. Chemie, Göttingen, Germany). All non-hydrogen atoms were refined with anisotropic displacement parameters. The hydrogen atoms

 Table 3:
 Crystal data and structure refinement data for complex 1.

Complex	1
Empirical formula	C44H36ClCu2N4NaO12
M _r	998.31
Crystal system	Monoclinic
Crystal size, mm ³	$0.14 \times 0.10 \times 0.09$
Space group	<i>P</i> 2 ₁ / <i>c</i> (No. 14)
<i>a</i> , Å	12.1098(5)
<i>b</i> , Å	17.6606(7)
<i>c</i> , Å	19.3747(7)
β , deg	92.119(2)
<i>V</i> , Å ³	4140.8(3)
Z	4
Т(К)	294(2)
μ (Mo <i>K</i> α), cm ⁻¹	1.2
$D_{\rm calcd.}$, g cm ⁻³	1.60
F(000), e	2040
heta range for data collection, deg	1.561-25.499
hkl ranges	$\pm 14, \pm 21, \pm 23$
Total/unique data/R _{int}	60832/7718/0.075
Observed data $[l > 2 \sigma(l)]$	4741
Ref. parameters	600
Final $R1/wR2$ indices $[I > 2 \sigma(I)]$	0.0495/0.1119
Final <i>R</i> 1/ <i>wR</i> 2 indices (all data)	0.0929/0.1343
Goodness-of-fit on F ²	1.017
$\Delta \rho$ (max/min), e Å ⁻³	-0.38/0.44

were placed geometrically and refined with isotropic thermal parameters using a riding model approximation. The perchlorate anion is disordered over two orientations sharing three oxygen atoms with refined occupancy ratio 0.524(10): 0.476(10). In order to prevent inflated anisotropy to the anisotropic displacement parameters, the Cl and O atoms of the disordered perchlorate anion were restrained to be approximately isotropic using the ISOR instruction in SHELXL-2014 [36, 37] (Institut Anorg. Chemie, Göttingen, Germany) with an effective standard deviation of 0.1² Å. All calculations and graphical illustrations for the complex have been performed using SHELXL-2014/7 [36, 37] (Institut Anorg. Chemie, Göttingen, Germany), and ORTEP [38, 39] programs (Oak Ridge National Laboratory, Oak Ridge, USA). Selected crystallographic data and important structure refinement parameters of 1 are summarized in Table 3.

CCDC 1501781 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

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Graphical synopsis

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