

# Synthesis and Crystal Structure of a New Hetero-nuclear $\text{Cu}_2\text{Sr}$ Complex of Salen Type Ligand

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## Abstract

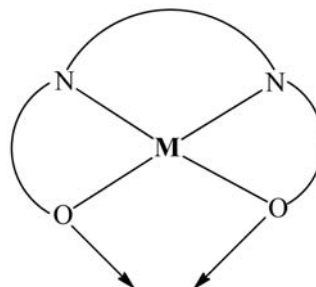
A new hetero-trinuclear complex  $[\text{Cu}_2(\mu\text{-L})_2\text{Sr}(\text{NO}_3)_2]$  has been synthesized and characterized by elemental analysis, IR, UV-Vis and X-ray crystallography, where L is a dianionic tetradentate Schiff base ligand with  $\text{N}_2\text{O}_2$  donor atoms. The X-ray structure indicates that the trinuclear complex adopts a bent structure for the three metal atoms, with a  $\text{Cu1-Sr-Cu2}$  intramolecular angle of approximately  $99.1^\circ$ . In the complex, the two terminal Cu ions are situated in a distorted square planar geometry coordinated by two imine nitrogen atoms and two phenolato oxygen atoms from the Schiff base ligand. The two neutral CuL complexes are bonded to a Sr(II) ion. The geometry of central strontium atom is a distorted square antiprism formed by eight oxygen atoms, four oxygen atoms belonging to the two Schiff base ligands from two units of CuL and four oxygen atoms belonging to the two bidentate nitrate ions. The two bidentate nitrates are cis to each other.

**Keywords:** Hetero-trinuclear complex, Schiff base, Copper(II) complexes, strontium ion, salicylaldehyde

## 1. Introduction

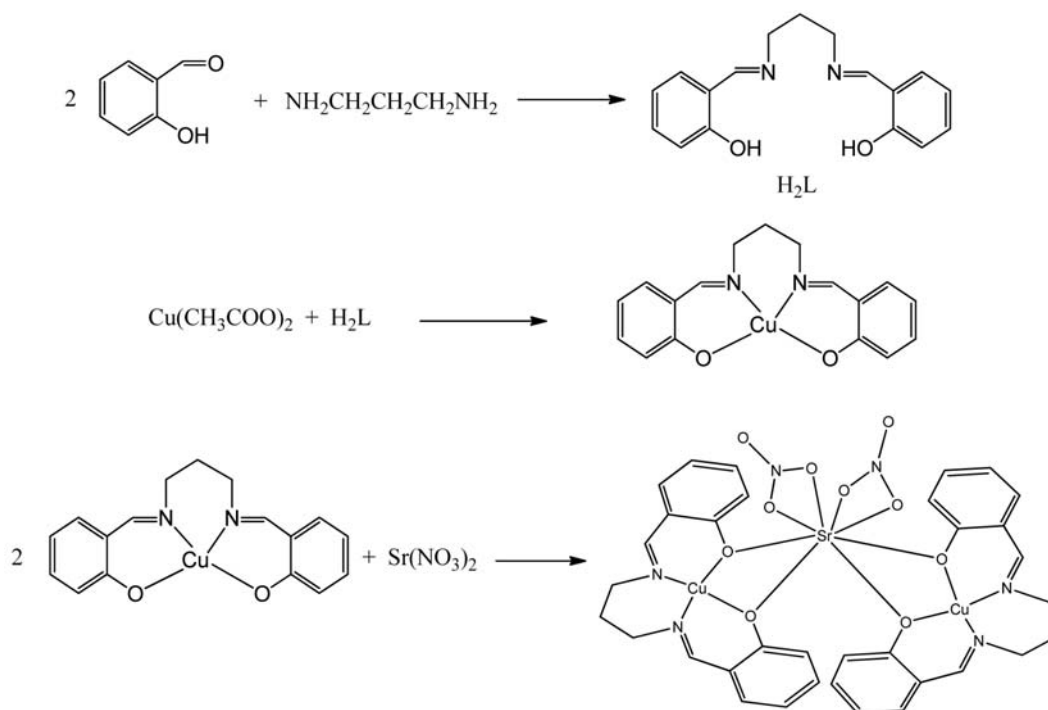
In the last few decades, one of the most prominent families of compounds in modern coordination chemistry are the poly-nuclearcopper(II) complexes of Schiff base ligands.<sup>1–6</sup> The complexes not only have played an important role in the development of modern coordination chemistry, but also they can utilize as model compounds at the active sites of multimetallo enzymes.<sup>7–10</sup> Investigations of hetero-nuclear complexes are more informative than those of homo-nuclear complexes, due to their interesting properties arising from different metal ion.<sup>11–15</sup>

Amongst the variety of methodologies applied to synthesize hetero-nuclear complexes, tetradentate Schiff base ligands containing imine nitrogen and alcoholic or phenolic oxygen donor atoms can be used. The internal  $\text{N}_2\text{O}_2$  cavity of the tetradentate Schiff base is a suitable complexation point for divalent metal ions; however, the Schiff base complexes can act as a bidentate chelating ligand for further coordination as a result of the interaction with other metal ions to form hetero-trinuclear complexes (Scheme 1), known as *ligand complexes*.<sup>16,17</sup>



Scheme 1. Ligand complexes

In the previous work, we reported synthesis, crystal structure and DFT analysis of the homo-trinuclear copper complex,  $\text{Cu}_3$ ,  $[\text{Cu}_2(\mu\text{-L})_2\text{CuCl}_2]$  (L = tetradentate Schiff base, salpn) complex.<sup>5</sup> In order to investigate the effect of the second metal on the structure of the trinuclear complexes, we have synthesized a hetero-trinuclear complex,  $\text{Cu}_2\text{Sr}$  with the salpn Schiff base ligand (Scheme 2). It has been observed that change in the central metal of the trinuclear complex (Cu with Sr) can cause change in the structure of the complexes.

Scheme 2. Synthesis hetero-trinuclear complex,  $\text{Cu}_2\text{Sr}$ 

## 2. Experimental

### 2.1. General

All chemicals were used as supplied by Merck and Fluka without further purification.

Infrared spectra were taken with an Equinox 55 Bruker FT-IR spectrometer using KBr pellets in the 400–4000  $\text{cm}^{-1}$  range. Absorption spectra were determined in the solvent methanol using GBC UV-Vis Cintra 101 spectrophotometer with 1 cm quartz, in the range of 200–800 nm at 25 °C. Elemental analyses (C, H, N) were performed by using a CHNS-O 2400II PerkinElmer elemental analyzer.

### 2.2. X-ray Crystallography

Diffraction images were measured on a Nonius Kappa CCD diffractometer (Mo K $\alpha$ , graphite monochromator,  $\lambda = 0.71073 \text{ \AA}$ ), and data were extracted using the DENZO/SCALEPACK package.<sup>18</sup> The structure solution was determined by direct methods (SIR92),<sup>19</sup> and refinement was performed using the CRYSTALS program package.<sup>20</sup> Atomic coordinates, bond lengths and angles and displacement parameters have been deposited at the Cambridge Crystallographic Data Centre. The crystallographic data for the complexes are summarized in Table 1.

### 2.3. Syntheses

#### 2.3.1. Synthesis of Schiff Base Ligand, $\text{H}_2\text{L}$

The tetradentate Schiff base  $\text{H}_2\text{L}$  was prepared by a general literature method,<sup>21–23</sup> by the condensation reaction

Table 1. Crystallographic data

Compound	$\text{Cu}_2(\mu\text{-L})_2\text{Sr}(\text{NO}_3)_2$
Chemical formula	$\text{C}_{34}\text{H}_{32}\text{Cu}_2\text{N}_6\text{O}_{10}\text{Sr}$
Formula weight	899.44
Temperature (K)	200
Space group	Triclinic, $P\bar{1}$ $Z = 2$
Unit cell dimensions	
a ( $\text{\AA}$ )	10.5177 (2)
b ( $\text{\AA}$ )	11.2970 (2)
c ( $\text{\AA}$ )	14.4353 (2)
$\alpha$ ( $^\circ$ )	86.9438 (10)
$\beta$ ( $^\circ$ )	80.1031 (10)
$\gamma$ ( $^\circ$ )	89.2079 (9)
V ( $\text{\AA}^3$ )	1687.23 (5)
F(000)	908
$D_{\text{calc}}$ ( $\text{g cm}^{-3}$ )	1.770
Crystal dimension (mm)	$0.43 \times 0.13 \times 0.05 \text{ mm}$
$\mu$ ( $\text{mm}^{-1}$ )	2.89
$\theta$ range for data collection ( $^\circ$ )	2.5–27.5
Limiting indices	$-13 \leq h \leq 13,$ $-14 \leq k \leq 14,$ $-18 \leq l \leq 18$
R[F2 > 2 $\sigma$ (F2)]	0.030
wR(F2) (all data)	0.073*

\*  $w = 1/[\sigma^2(F^2) + (0.04P)^2 + 1.63P]$ , where  $P = (\max(F_o^2, 0) + 2F_c^2)/3$ 

between 2 equivalents of salicylaldehyde and 1 equivalent of 1,3-propanediamine in methanol. The resulting bright yellow solution was heated to reflux for 1 h. Upon cooling, a yellow product was obtained. The yield was 70%.

### 2. 3. 2. Synthesis of the CuL Complex

The copper(II) complex was prepared by a general method,<sup>21,24</sup> using the reaction between  $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$  and the Schiff base ligand,  $\text{H}_2\text{L}$ , (1:1 ratio) in methanol. The resulting solution was heated to reflux for 1 h. The resulting green precipitates were collected by filtration and washed with methanol. The yield was 50%. Anal. Calcd. for  $\text{C}_{17}\text{H}_{16}\text{CuN}_2\text{O}_2$  (%): C, 59.38; H, 4.69; N, 8.15. Found: C, 59.12; H, 4.63; N, 8.21. IR (KBr,  $\text{cm}^{-1}$ ):  $\nu_{\text{C}=\text{N}} = 1607$ ,  $\nu_{\text{C}-\text{O}} = 1327$ . Electronic spectra for  $\text{CH}_3\text{OH}$ : d-d,  $\lambda_{\text{max}}$  (log  $\epsilon$ ) 607 nm (2.39).

### 2. 3. 3. Syntheses of $[\text{Cu}_2(\mu\text{-L})_2\text{Sr}(\text{NO}_3)_2]$ , $\text{Cu}_2\text{Sr}$

The CuL complex (0.344 g, 1 mmol) was dissolved in 50 mL methanol and warmed. About 10 mL of methanolic solution of  $\text{Sr}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  (0.105 g, 0.5 mmol) was added to it. The resulting mixture was then refluxed for 4 h. The resulting green precipitates were collected by filtration and recrystallized from methanol/toluene (1:1 v/v) olive-green laths crystals appeared upon slow evaporation of the solvents. Yield 60%. Anal. Calcd. for  $\text{C}_{34}\text{H}_{32}\text{Cu}_2\text{SrN}_6\text{O}_{10}$  (%): C, 45.44; H, 3.59; N, 9.36. Found: C, 44.20; H, 3.40; N, 9.43. IR (KBr,  $\text{cm}^{-1}$ ):  $\nu_{\text{C}=\text{N}} = 1618$ ,  $\nu_{\text{C}-\text{O}} = 1316$ . Electronic spectra for  $\text{CH}_3\text{OH}$ : d-d,  $\lambda_{\text{max}}$  (log  $\epsilon$ ) 607 nm (2.65).

## 3. Results and Discussion

### 3. 1. Synthesis

The hetero-trinuclear metal complex,  $\text{Cu}_2\text{Sr}$  was synthesized in two steps, reaction of  $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$  and  $\text{H}_2\text{L}$  in 1:1 molar ratio under reflux in methanol gave CuL complex. Then it was mixed with strontium salt in 2:1 molar ratio in methanol to form the hetero-trinuclear metal complex  $[\text{Cu}_2(\mu\text{-L})_2\text{Sr}(\text{NO}_3)_2]$ ,  $\text{Cu}_2\text{Sr}$ .

The suitable crystals for X-ray diffraction study were obtained from methanol/toluene (1:1 v/v), by slow evaporation of solvents.

The purity of the complex was checked by C, H, N elemental analyses which led to our postulated  $[(\text{CuL})_2\text{Sr}(\text{NO}_3)_2]$  empirical formula for this complex.

### 3. 2. Description of the Crystal Structure

The solid state structure of the hetero-trinuclear complex of  $[\text{Cu}_2(\mu\text{-L})_2\text{Sr}(\text{NO}_3)_2]$  obtained by single-crystal X-ray diffraction is shown in Figs. 1 and 2. The selected main bond lengths and angles are summarized in Table 2.

The unit cell contains two distinct entities of  $[\text{Cu}_2(\mu\text{-L})_2\text{Sr}(\text{NO}_3)_2]$  moieties. In the complex, the two terminal Cu ions are four coordinate with distorted square planar geometry by two imine nitrogen atoms and two phenolato oxygen atoms belonging to the Schiff base ligand.

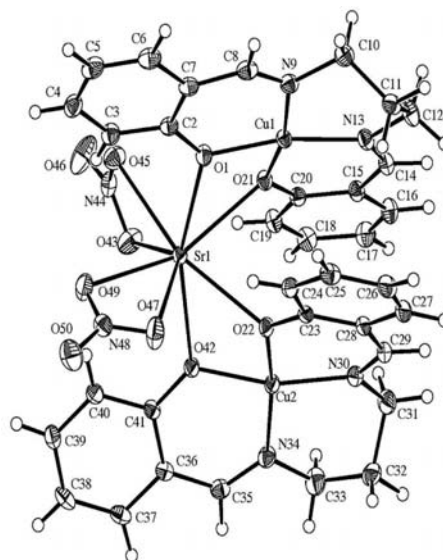


Fig. 1. The molecular structure of  $[\text{Cu}_2(\mu\text{-L})_2\text{Sr}(\text{NO}_3)_2]$  complex with labelling of selected atoms.

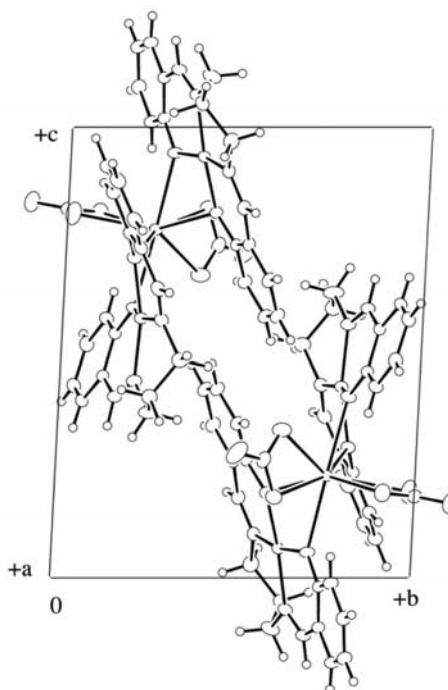


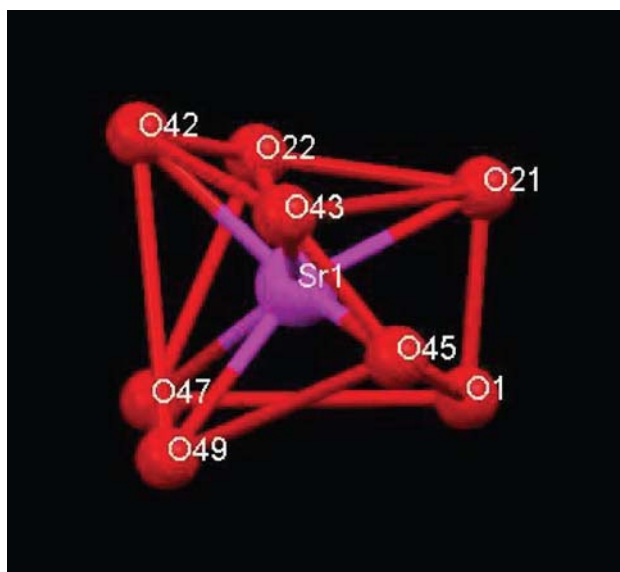
Fig. 2. Unit cell packing diagram of  $[\text{Cu}_2(\mu\text{-L})_2\text{Sr}(\text{NO}_3)_2]$  complex projected down the  $c$  axis.

Whereas in  $\text{Cu}_3$  complex, the coordination geometry around the two terminal copper(II) ions are five coordinate with a distorted square pyramide geometry.<sup>5</sup> The two neutral CuL complexes are bonded to a Sr(II) ion. The geometry of trinuclear complex  $\text{Cu}_2\text{Sr}$  is a bent structure with a  $\text{Cu}(1)\text{-Sr}(1)\text{-Cu}(2)$  intra-atomic angle of approximately  $99.1^\circ$  and is larger than the value observed for  $\text{Cu}_3$  complex ( $90.62(2)^\circ$ ).<sup>5</sup> This could be due to the fact that in hetero-trinuclear complex,  $\text{Cu}_2\text{Sr}$  radii of strontium ion is larger than radii of copper ion.

**Table 2.** Selected bond lengths (Å) and angles (°) in [Cu<sub>2</sub>(μ-L)<sub>2</sub>Sr(NO<sub>3</sub>)<sub>2</sub>] complex

Cu1–O1	1.9476 (16)	Sr1–O1	2.5662 (15)
Cu1–O21	1.9403 (15)	Sr1–O21	2.5446 (15)
Cu1–N9	1.9739 (19)	Sr1–O22	5.5185 (15)
Cu1–N13	1.9887 (19)	Sr1–O42	2.5962 (15)
Cu2–O22	1.9192 (15)	Sr1–O43	2.6435 (19)
Cu2–O42	1.9404 (16)	Sr1–O45	2.6841 (19)
Cu2–N30	1.9740 (20)	Sr1–O47	2.6370 (20)
Cu2–N34	1.9760 (20)	Sr1–O49	2.6012 (19)
O1–Cu1–O21	79.78 (6)	O22–Sr1–O43	122.11 (6)
O1–Cu1–N9	92.11 (8)	O42–Sr1–O43	71.99 (6)
O21–Cu1–N9	167.31 (7)	O1–Sr1–O45	78.39 (5)
O1–Cu1–N13	169.26 (7)	O21–Sr1–O45	88.10 (5)
O21–Cu1–N13	92.61 (7)	O22–Sr1–O45	157.62 (6)
N9–Cu1–N13	96.62 (8)	O42–Sr1–O45	119.10 (5)
O22–Cu2–O42	81.75 (6)	O43–Sr1–O45	47.52 (6)
O22–Cu2–N30	91.83 (7)	O1–Sr1–O47	92.20 (7)
O42–Cu2–N30	165.39 (8)	O21–Sr1–O47	130.99 (6)
O22–Cu2–N34	166.56 (8)	O22–Sr1–O47	75.93 (6)
O42–Cu2–N34	92.24 (8)	O42–Sr1–O47	85.24 (7)
N30–Cu2–N34	96.63 (8)	O43–Sr1–O47	130.91 (7)
O1–Sr1–O21	58.40 (5)	O1–Sr1–O49	97.57 (6)
O1–Sr1–O22	100.66 (5)	O21–Sr1–O49	155.21 (6)
O21–Sr1–O22	72.96 (5)	O22–Sr1–O49	121.65 (5)
O1–Sr1–O42	159.69 (5)	O42–Sr1–O49	95.72 (6)
O21–Sr1–O42	109.06 (5)	O43–Sr1–O49	90.51 (7)
O22–Sr1–O42	59.16 (5)	O45–Sr1–O47	126.35 (6)
O1–Sr1–O43	123.03 (6)	O45–Sr1–O49	80.37 (6)
O21–Sr1–O43	97.80 (6)	O47–Sr1–O49	48.30 (6)

The central strontium ion is eight coordinate with distorted square anti-prism geometry (Fig. 3) with four oxygen atoms belonging to the two Schiff base ligand from two units of CuL (O1, O21, O22 and O42) and four

**Fig. 3.** View of the coordination environment around strontium center.

oxygen atoms belonging to the two bidentate nitrate ions (O43, O45, O47 and O49). The Sr–O distances range between 2.5185(5)–2.6841(19) Å. Four of the bond lengths belonging to two CuL units are shorter than four of nitrate ions. The NO<sub>3</sub><sup>−</sup> groups have weak basicity, and bind weakly to Sr ion as a bidentate ligand.

The two bidentate nitrates are cis to each other. The N–O bond lengths in two nitrate ions are nearly equal, which are in agreement with the bidentate nature of NO<sub>3</sub><sup>−</sup> ligand.<sup>25</sup>

As shown in Table 2, the average bond lengths of Cu–N are 1.9713(19) Å and 1.975(2) Å for the two terminals CuL moieties. The bond average lengths for the Cu–O entities are 1.9439(16) Å and 1.9298(16) Å. These are similar to values observed for Cu<sub>3</sub>.<sup>5</sup> These Cu–O and Cu–N distances in two CuL units are slightly greater than the average Cu–O and Cu–N distances, 1.8965(5) and 1.9705(6) Å, respectively, in the mononuclear CuL complex.<sup>21</sup> These increases in the bond lengths have been also observed in polynuclear complexes in comparison to the mononuclear complex in similar systems.<sup>4,5</sup>

The average O–Cu–N trans angles in the two terminal units are 168.29(7) and 165.98(8). These are similar to values reported for Cu<sub>3</sub> complex (163.57(2) and 164.98(2)°), indicating that there are tetrahedral distortion from the ideal square planar geometry.

The molecular packing in the solid state is formed by van der Waals interactions mainly without both H-bonds and π–π stacking interactions. The distance between aromatic moieties of Schiff base ligands are too great for any significant π–π interactions within the crystal lattice.

### 3. 3. Infrared Spectra

The strong ν(C=N) band at 1606 cm<sup>−1</sup> for CuL is shifted considerably toward lower energy in comparison with the H<sub>2</sub>L ligand (1631 cm<sup>−1</sup>), which indicates the coordination of the imine nitrogen atom to the metal center.<sup>2,26</sup> Conversely, the ν(C–O) band is shifted approximately 40 cm<sup>−1</sup> toward higher energies compared to the free ligand, which suggests the coordination of the deprotonated phenolic oxygen atoms to the copper center in the complex.<sup>27,28</sup> The ν(C=N) shifts by about 10 cm<sup>−1</sup> to higher energy in the trinuclear complex compared to the CuL complex. This hypsochromic shift of the ν(C=N) for the trinuclear complex relative to the CuL complex is likely explained in terms of a weaker copper-ligand coordinative interaction.

The ν(C–O) in the two terminals moieties in the trinuclear complex that appear at 1316 cm<sup>−1</sup> are shifted to lower energy relative to the CuL complex. These shifts of the ν(C=N) (to higher energy) and the ν(C–O) (to lower energy) in trinuclear complexes relative to the mononuclear CuL complex are explained in terms of a weaker Cu–ligand coordinative interaction.

## 4. Conclusion

The new hetero-trinuclear complex  $[\text{Cu}_2(\mu\text{-L})_2\text{Sr}(\text{NO}_3)_2]$ ,  $\text{Cu}_2\text{Sr}$  has been synthesized and the crystal structure of the complex has been determined. In complex  $\text{Cu}_2\text{Sr}$ , the two terminal Cu ions are four coordinate with distorted square planar geometry and the strontium environment is eight coordinate with distorted square antiprism. The hetero-trinuclear complex has bent structure.

## 5. Supplementary Data

CCDC 866027 contains the supplementary crystallographic data for  $[\text{Cu}_2(\mu\text{-L})_2\text{Sr}(\text{NO}_3)_2]$ . These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk).

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## Povzetek

Sintetizirali smo nov hetero-trinuklearni kompleks  $[\text{Cu}_2(\mu\text{-L})_2\text{Sr}(\text{NO}_3)_2]$ , kjer je ligand L dianionska tetradentatna Schiffova baza z  $\text{N}_2\text{O}_2$  donorskimi atomi. Koordinacijsko spojino smo okarakterizirali z elementno analizo, IR in UV-Vis spektroskopijo in rentgensko strukturno analizo. Kristalna struktura kaže, da ima trinuklearni kompleks kotno strukturo treh kovinskih atomov s kotom Cu1–Sr–Cu2 približno  $99.1^\circ$ . Dva terminalna bakrova iona sta koordinirana z dvema iminskima dušikovima atomoma in dvema fenolatinima kisikovima atomoma Shiffove baze s popačeno kvadratno-planarno geometrijo. Dve nevtralni CuL enoti sta vezani na centralni Sr(II) ion. Geometrija centralnega stroncijevega atoma je popačena kvadratna antiprizma, ki jo tvori osem kisikovih atomov, štirje pripadajo dvema Shiffovima bazama iz dveh CuL enot, drugi štirje kisikovi atomi pa dvema bidentatno koordiniranim nitratnim ionoma. Bidentatna nitratna iona sta koordinirana v *cis* položaju.