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### Synthesis, characterization and crystal structure of a CuSCN complex containing a new bidentate *N,N*-bis(2-nitrocinnamaldehyde)ethylenediamine Schiff base and triphenylphosphine

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# Synthesis, characterization and crystal structure of a CuSCN complex containing a new bidentate *N,N'*-bis(2-nitrocinnamaldehyde)ethylenediamine Schiff base and triphenylphosphine

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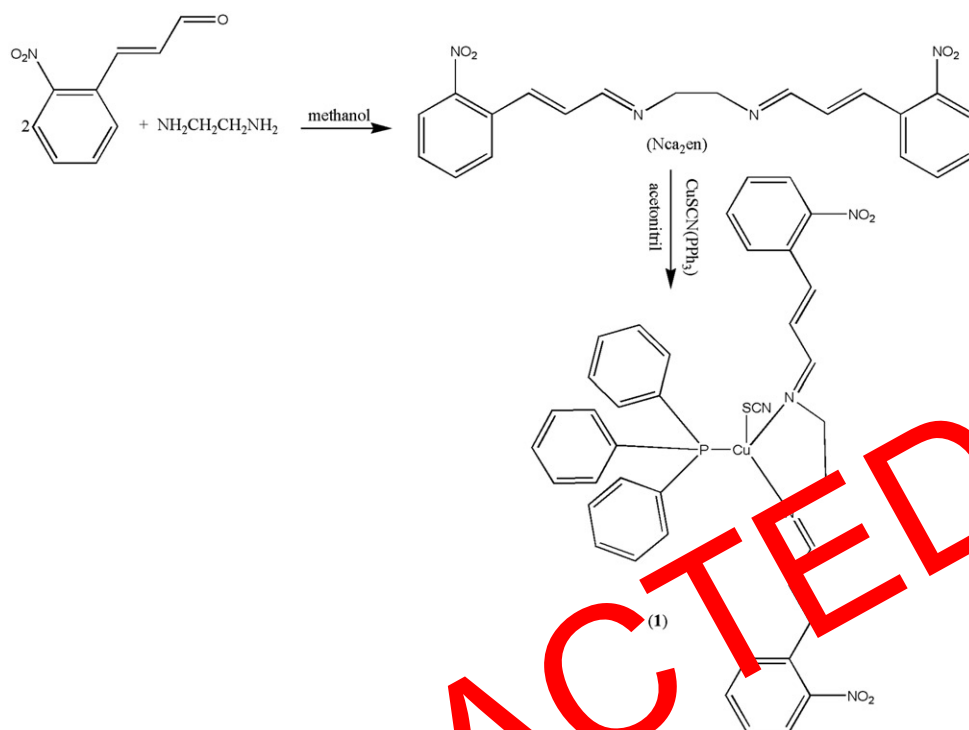
Reaction of copper(I)thiocyanate (CuSCN) and triphenylphosphine (PPh<sub>3</sub>) with a new bidentate Schiff base, *N,N'*-bis(2-nitrocinnamaldehyde)ethylenediamine (*Nca<sub>2en</sub>*), in a 1:1 mol ratio in acetonitrile resulted in the formation of a complex formulated as [CuSCN(*Nca<sub>2en</sub>*)(PPh<sub>3</sub>)]. The ligand (*Nca<sub>2en</sub>*) and complex were characterized by analyses, IR, electronic and UV-IR spectroscopy, and X-ray diffraction. The *Nca<sub>2en</sub>* is roughly planar and displays a *trans* configuration with respect to the C=N double bond. *Nca<sub>2en</sub>* acts as a bidentate ligand coordinating via two N atoms to copper and adopts a *trans, trans* configuration in this complex. The SCN ligand is coordinated through sulfur. The ligand and its copper(I) complex crystallize in the monoclinic space group. The geometry around the copper atom is distorted tetrahedral with  $\angle(3)\text{-Cu-N}(2)$ , 81.59(11) and  $\text{P-Cu-S}$ , 117.29(9).

**Keywords:** Schiff base; Copper(I) complex; Tetrahedral complex; Crystal structures

## 1. Introduction

Transition metal compounds containing Schiff-base ligands [1–7] play an important role in coordination chemistry related to catalysis and enzymatic reactions, magnetism, and molecular architectures [8, 9]. Coordination chemistry of copper(I) has application in catalytic processes, photosensitization, light harvesting, and the design of supramolecular arrays [10–14]. Steric, electronic, and conformational effects imparted by the coordinated ligand play an important role in modifying properties of metal complexes [15, 16]. In this research we report synthesis, characterization, and structures of *N,N'*-bis(2-nitrocinnamaldehyde)ethylenediamine (*Nca<sub>2en</sub>*) and its Cu(I) complex (scheme 1).

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Scheme 1. Synthesis of *Nca*<sub>2</sub>*en* and [CuSCN(*Nca*<sub>2</sub>*en*)(PPh<sub>3</sub>)] (1).

## 2. Experimental

### 2.1. Materials and physical measurements

All chemicals were purchased from commercial sources and used as received. Infrared spectra were obtained with a Shimadzu IR-435 spectrophotometer using KBr pellets. Visible absorption spectrum was recorded with a Shimadzu 160 spectrophotometer. NMR spectra were recorded on a Bruker AW 400 MHz. Analyses for C, H, and N were determined on a Carlo Erba 1108 Elemental Vario EL analyzer.

### 2.2. Synthesis of *Nca*<sub>2</sub>*en*

To a solution of 60 mg (1 mmol) ethylenediamine in 10 mL methanol, cooled in an ice bath, 354 mg (2 mmol) of 2-nitrocinnamaldehyde was added dropwise. The mixture was then stirred for an additional 1 h. *Nca*<sub>2</sub>*en* was obtained as a white microcrystalline precipitate which was filtered off and washed with 5 mL cold absolute methanol. The crude product was recrystallized from ethanol to give white needle-shaped crystals. The yield was 340 mg, 90%. m.p. 175–177°C. IR (KBr pellet, 400–4000 cm<sup>-1</sup>) 1632 (C=N). <sup>1</sup>H NMR (CDCl<sub>3</sub>) 3.91 (s, 4H, -CH<sub>2</sub>-CH<sub>2</sub>-), 7.5 (d, 2H<sub>c</sub>), 6.89 (dd, 2H<sub>b</sub>), 8.01 (d, 2H<sub>d</sub>), 7.44–7.81(m, 8H, ArH), 8.01 (d, 2H<sub>a</sub>, -CH=N). Anal. Calcd for C<sub>20</sub>H<sub>18</sub>N<sub>4</sub>O<sub>4</sub>: C, 63.48; H, 4.76; N, 14.81%. Found: C, 63.36; H, 4.71; N, 14.76%.

Table 1. Data collection and structure refinement parameters for *Nca<sub>2</sub>en* and **1**.

Chemical formula	C <sub>20</sub> H <sub>18</sub> N <sub>4</sub> O <sub>4</sub>	C <sub>39</sub> H <sub>33</sub> CuN <sub>5</sub> O <sub>4</sub> PS
Formula weight	378.38	762.27
Crystal color	Colorless	Orange
Temperature (K)	120(2)	293(2)
$\lambda$ (Å)	0.67270	0.71073
Crystal system	Monoclinic	Triclinic
Crystal size (mm <sup>3</sup> )	0.10 × 0.08 × 0.01	0.34 × 0.30 × 0.30
Space group (Å, °)	<i>P</i> 2 <sub>1</sub> / <i>c</i>	<i>P</i> 1
<i>a</i>	11.439(2)	9.1038(8)
<i>b</i>	3.7910(8)	13.8186(12)
<i>c</i>	20.541(5)	15.2972(14)
$\alpha$	90	104.7830(10)
$\beta$	97.43(3)	102.6780(10)
$\gamma$	90	91.3000(10)
<i>V</i> (Å <sup>3</sup> )	883.3(3)	883.3(3)
<i>Z</i>	2	2
<i>D</i> <sub>Calcd</sub> (g cm <sup>-3</sup> )	1.423	1.399
$\mu$ (mm <sup>-1</sup> )	0.102	0.754
<i>F</i> (000)	396	788
Number of reflections collected	12,986	11,222
Number of independent reflections ( <i>R</i> <sub>int</sub> )	13,147 (0.0000)	6371 (0.0336)
Number of reflections used [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ]	10,186	590
Final <i>R</i> indices [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ]	<i>R</i> <sub>1</sub> = 0.0705, <i>wR</i> <sub>2</sub> = 0.1959	<i>R</i> <sub>1</sub> = 0.0590, <i>wR</i> <sub>2</sub> = 0.1032
Final <i>R</i> indices (all data)	<i>R</i> <sub>1</sub> = 0.0859, <i>wR</i> <sub>2</sub> = 0.2049	<i>R</i> <sub>1</sub> = 0.0775, <i>wR</i> <sub>2</sub> = 0.1048

### 2.3. Synthesis of [CuSCN(*Nca<sub>2</sub>en*)(PPh<sub>3</sub>)] (**1**)

To a solution of 121 mg (1 mmol) CuSCN in 5 mL acetonitrile a solution of 261 mg (1 mmol) PPh<sub>3</sub> in 5 mL acetonitrile was added. The mixture was stirred for 5 min and then 37 mg (1 mmol) of *Nca<sub>2</sub>en* in 5 mL acetonitrile was added and stirred for an additional 5 min. The volume of the solvent was reduced under vacuum to about 5 mL. Diffusion of diethyl ether vapor into the concentrated solution gave needle-like orange crystals suitable for X-ray studies. The crystals were collected and dried *in vacuo*. The yield was 611 mg, 80%. IR (KBr pellet, 400–4000 cm<sup>-1</sup>) 1620 (C=N), 2010 (NCS). <sup>1</sup>H NMR (CDCl<sub>3</sub>) 3.82 (s, 4H, –CH<sub>2</sub>–CH<sub>2</sub>–), 7.21–7.98 (m, 27H, Ph–CH=C, C=CH–C, ArH), 8.11 (d, 2H<sub>A</sub>, –CH=N–). Anal. Calcd for C<sub>39</sub>H<sub>33</sub>CuN<sub>5</sub>O<sub>4</sub>PS: C, 61.45; H, 4.33; N, 9.19%. Found: C, 61.41; H, 4.30; N, 9.13%.

### 2.4. X-ray diffraction analysis

Single crystal diffraction studies were carried out on a Bruker SMART 1K CCD diffractometer with Mo-K $\alpha$  (0.71073 Å) radiation. The structures were solved by direct methods and the positional and anisotropic displacement parameters of the non-hydrogen atoms were refined by a least-squares calculation on *F*<sup>2</sup>. A summary of the crystal data along with further details of the structure determination and refinement are given in table 1. Unit cell parameters were determined using SMART [17] and refined based on the positions of all strong reflections using SAINT [17]. Absorption correction was by SADABS [18] based on symmetry-equivalent and repeated reflections. The structure was solved by direct methods using SIR97 [19] and refined by full matrix

least-squares on  $F^2$  using SHELXTL [20]. Molecular graphics were produced using DIAMOND-3 [21] and Mercury 1.4 [22].

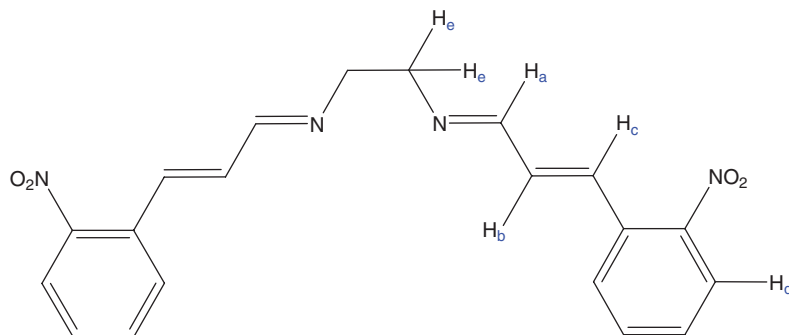
### 3. Results and discussion

#### 3.1. Spectral characterization

The IR spectrum of the free ligand exhibits the characteristic band of an imine at  $1630\text{ cm}^{-1}$ . This band shifts to lower frequencies in the IR spectra of complex due to coordination of the imine nitrogen [14]. A strong band at  $2070\text{ cm}^{-1}$  and a weak band at  $782\text{ cm}^{-1}$  are assigned to the S-bonded SCN in this complex [23].  $^1\text{H}$  NMR data of *Nca*<sub>2</sub>*en* suggest that the ligand has a symmetrical structure (scheme 2). The four methylene protons are a singlet at 3.91 ppm. The two vinyl CH protons ( $\text{H}_b$ ) are observed as a doublet of doublets centered at 6.89 ppm,  $\text{H}_d$  is observed as a doublet centered at 8.01 ppm and the multiplet centered 7.44–7.81 ppm is assigned to the phenyl protons. Two protons ( $\text{H}_c$ ) are observed as a doublet centered at 7.5 ppm. The  $\text{CH}=\text{N}$  protons ( $\text{H}_a$ ) are a doublet at 8.78 ppm. The  $^1\text{H}$  NMR peak assignments are in the experimental section for  $[\text{CuSCN}(\text{Nca}_2\text{en})(\text{PPh}_3)]$ . The proton resonances of coordinated *Nca*<sub>2</sub>*en* are commonly observed, however, the aromatic protons of the coordinated  $\text{PPh}_3$  ligands overlap to some extent with those of the phenyl protons of *Nca*<sub>2</sub>*en*. Aside from the aromatic protons which appear at about 7.21–7.98 ppm, the two imine protons appear as a doublet at about 8.78 ppm. The ethylenic protons overlap with those of the aromatic region. The downfield shift of the iminic protons relative to the free ligand can be attributed to the deshielding resulting from coordination of the ligand. The sharp singlet at 3.82 ppm is assigned to the  $\text{CH}_2\text{--CH}_2$  protons. In the UV–Vis spectrum of the Schiff-base ligand, the aromatic bands at 210–302 nm are attributed to a  $\pi \rightarrow \pi^*$  transition. The electronic spectrum of the Cu(I) complex shows an absorption at 283 nm attributed to the  $\pi \rightarrow \pi^*$  transition of the ligand.

#### 3.2. Crystal and molecular structure of *Nca*<sub>2</sub>*en*

The single-crystal X-ray structure determination of *Nca*<sub>2</sub>*en* was carried out at 120 K. Table 1 contains crystal parameters, data collection and refinement. Selected bond



Scheme 2. Symmetrical structure with protons position of *Nca*<sub>2</sub>*en*.

lengths, angles and torsion angles are listed in table 2. The structure analysis showed that the compound forms in monoclinic space group,  $P2_1/c$  with  $a=11.439(2)$ ,  $b=3.791(8)$ ,  $c=20.541(5)$  Å and  $\alpha=90$ ,  $\beta=97.43(3)$ , and  $\gamma=90$  with  $Z=2$ . In figure 1, two 2-nitrocinnamaldehyde groups are bridged by ethane-1,2-diamine via two C=N double bonds in a roughly linear geometry. The molecule is on the crystallographic center of symmetry. The torsion angles,  $N1'-C1'-C1-N1$  and  $C1-N1-C2-C3$ , are 180 and  $-178.78^\circ$ , respectively. The  $N1-C1$  and  $N1-C2$  bond lengths are 1.462 and 1.453 Å, respectively, which are typical of C-N and C=N bonds, respectively. The molecule of *N,N'*-bis(2-nitrocinnamaldehyde)ethylenediamine (*Nca<sub>2en</sub>*) is roughly planar and displays a *trans* configuration with respect to the C=N double bond. The distance between the planes of the benzene rings is 1.440 Å. In the crystal packing, the molecules are linked via weak inter and intramolecular  $H\cdots O-N$  and  $C-H\cdots N$  hydrogen bonds (figure 2). Intermolecular hydrogen bonds occur

Table 2. Selected bond lengths (Å) and angles ( $^\circ$ ).

<i>NcaR<sub>2en</sub></i>		[Cu <sub>2</sub> (CN)(Nca <sub>2en</sub> )(PPh <sub>3</sub> )]	
<b>Bond length (Å)</b>		<b>Bond length (Å)</b>	
O1–N2	1.3822(14)	Cu–S	2.016(11)
O2–N2	1.1943(11)	Cu–N3	2.072(7)
N1–C1	1.4619(12)	Cu–N2	2.105(7)
N1–C2	1.4534(15)	Cu–P	2.205(2)
N2–C10	1.3300(13)	S–C39	1.565(8)
C2–C3	1.380(14)	S–C39	1.177(12)
<b>Bond angle (<math>^\circ</math>)</b>		<b>Bond angle (<math>^\circ</math>)</b>	
C1–N1–C2	124.77(9)	S–Cu–N3	105.5(3)
O1–N2–O2	124.46(9)	S–Cu–N2	110.0(3)
O1–N2–C10	122.17(8)	N3–Cu–N2	81.6(3)
O2–N2–C10	104.87(10)	S–Cu–P	117.3(2)
N1–C2–C3	128.68(9)	C9–N2–Cu	134.5(7)
N2–C10–C9	123.59(9)	C39–S1–Cu	168.9(9)
N2–C10–C9	102.50(10)	S–C39–N5	178.6(11)
<b>Torsion angle (<math>^\circ</math>)</b>		<b>Torsion angle (<math>^\circ</math>)</b>	
C1–N1–C2–C3	127.11(12)	S–Cu–P–C21	82.4(4)
N1–N1–C2–C3	178.79(9)	N3–Cu–P–C21	–150.1(4)
N1–C2–C3–C4	–176.98(9)	S–Cu–N2–C10	131.6(6)
C2–C3–C4–C5	178.91(10)	Cu–S–C39–N5	174(100)

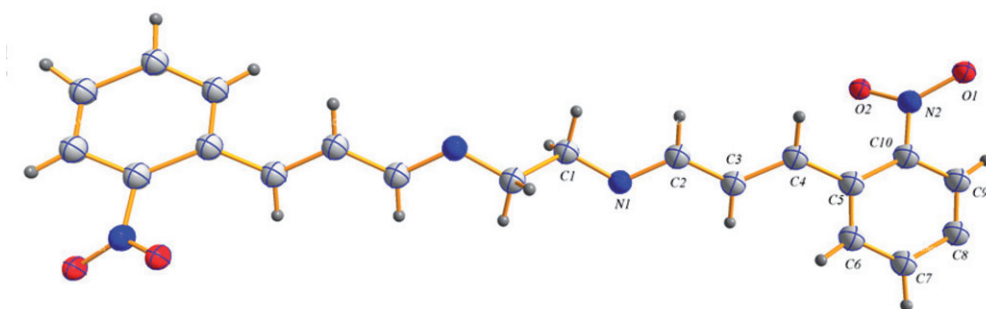
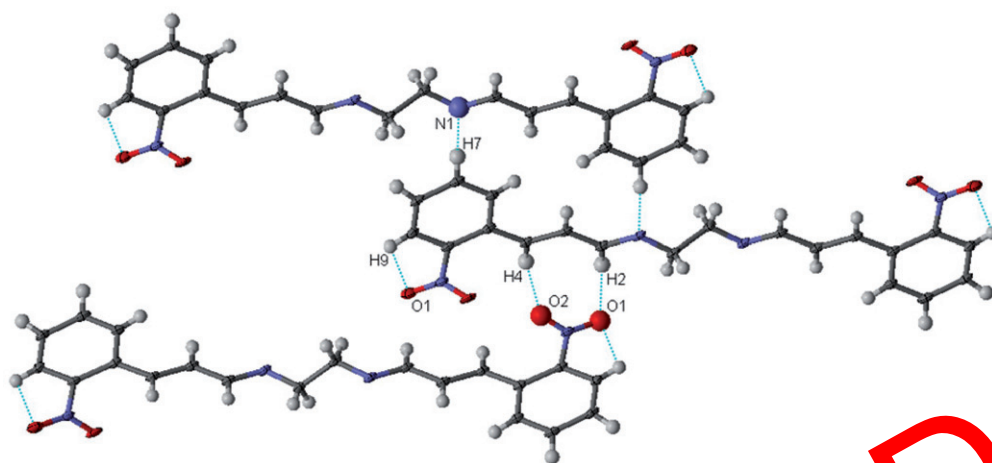


Figure 1. ORTEP structure of *Nca<sub>2en</sub>* showing the thermal ellipsoids at 50% probability.

Figure 2. The C–H...N and C–H...O hydrogen bonds of *Nca-en*.Table 3. Hydrogen-bonding geometry (Å, °) for *Nca-en*.

D–H...A	D–H	H...A	D...A	D–H...A
N2–O2...H4 <sup>i</sup>	1.194(1)	2.159(1)	3.119(19)	139.65(1)
N2–O1...H2 <sup>i</sup>	1.382(1)	2.672(1)	2.934(19)	87.90(1)
N2–O1...H9	1.382(1)	2.207(1)	2.340(19)	77.84(1)
C7–H7...N1 <sup>ii</sup>	0.951(1)	2.84(1)	3.193(19)	159.82(1)

Symmetry code: (i) 1–x, –y, 1–z; (ii) 1–x, 1–y, –z.

between H4 and O2 (2.159(1) Å) and between H2 and O1 (2.662(1) Å), H7 and N1 (2.84(1) Å) (table 3).

### 3. Crystal and molecular structure of [CuSCN(*Nca<sub>2</sub>en*)(PPh<sub>3</sub>)] (1)

This complex crystallizes in the triclinic crystal system with space group *P1*. The molecular structure and atom numbering scheme is illustrated in figure 3. Selected bond lengths and angles are given in table 2. The pseudohalide, SCN, known to coordinate in both terminal and bridging modes, is terminal in **1**. The structure consists of discrete four-coordinate [Cu(*Nca<sub>2</sub>en*)(PPh<sub>3</sub>)(SCN)] containing the bidentate Schiff base, PPh<sub>3</sub>, and SCN. The geometry around Cu(I) is distorted from tetrahedral to pseudotetrahedral. The ligand adopts a *trans, trans* configuration in this complex; the N2–Cu–N3 angle in this complex is only 81.6(3)°. The S1–Cu–P angle is 117.3(2)°, large for a tetrahedron. The dihedral angle between N3–Cu–N2 and S–Cu–P, 88.13, is indicative of deviation from idealized tetrahedron. The average Cu–N (*Nca<sub>2</sub>en*) distances of 2.0885 is similar to a pseudotetrahedral (diimine)copper(I) complex [24]. The mode of coordination of the thiocyanate to transition metals is influenced by electronic and steric effects of ancillary ligands as well as the solvents used for the synthesis and crystallization of the complexes [25, 26]. The terminal isothiocyanato in **1** is almost linear, S–C39–N5 = 178.6(11)°. Despite the fact that the donor nitrogen atoms

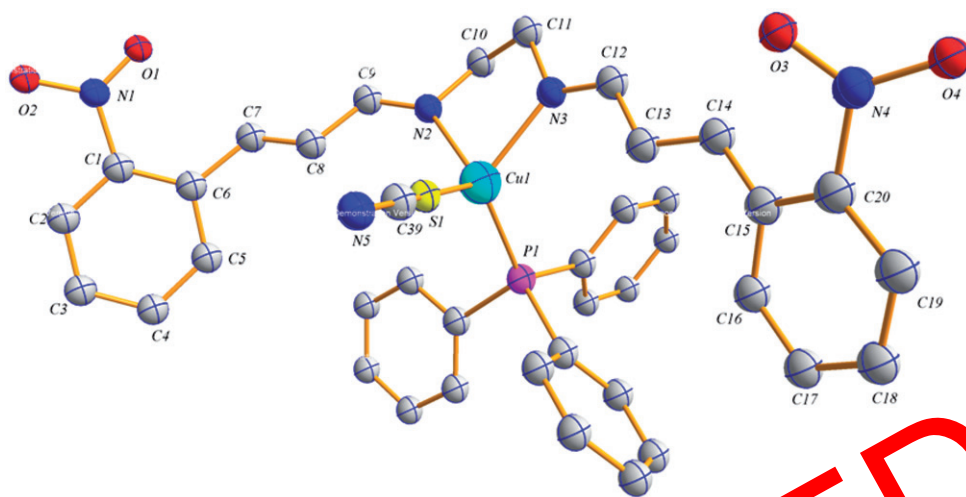


Figure 3. ORTEP structure of **1** showing the thermal ellipsoids at 50% probability. All H atoms have been omitted for clarity.

Table 4. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ) for

D–H...A	D–H	H...A	D...A	D–H...A
N1–O1...H14 <sup>i</sup>	1.201(1)	2.477(1)	3.888(19)	153.12(1)
N4–O3...H7 <sup>i</sup>	1.229(1)	2.521(1)	3.407(19)	125.68(1)
N1–O2...H2	1.251(1)	2.303(1)	2.558(19)	86.75(1)
N4–O4...H19	1.232(1)	2.515(1)	2.515(19)	80.09(1)
C38–H38...N3	0.951(1)	2.740(1)	3.610(19)	152.49(1)

Symmetry code: (i)  $x, 1-y, 1-z$ .

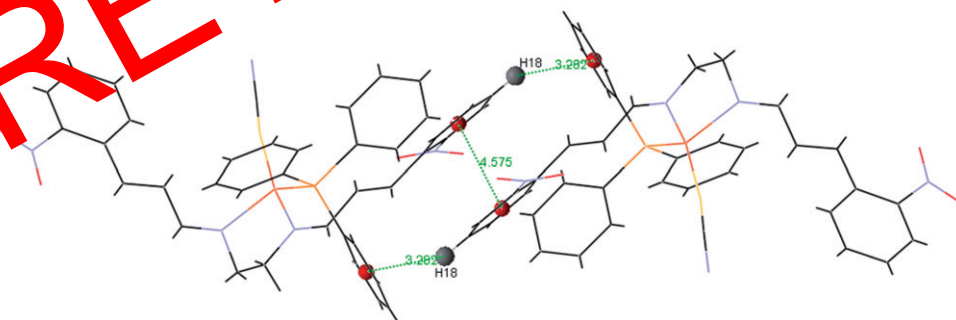


Figure 4. The stacking of **1** via  $\pi$ - $\pi$ , C–H... $\pi$  interactions.

in *Ncazen* are  $sp^2$  hybridized, the chelate ring is significantly puckered in this complex and some strain in the chelate ring is suggested by the deviation from the  $120^\circ$  angle about nitrogen: C9–N2–Cu and C10–N2–Cu,  $134.5(3)$  and  $105.9(2)$ . The single bond distance of C8–C9 in this complex,  $1.442(5)$ , slightly shorter than C10–C11,  $1.515(5)$ , indicates electron delocalization. Molecules are linked via weak inter and intramolecular C–H...O and C–H...N hydrogen bonds. Intermolecular hydrogen



bonds occur between H14 and O1 (2.476(1) Å) and between H7 and O3 (2.541(1) Å) (table 4). The benzene rings of **1** are stacked via  $\pi$ - $\pi$  interactions, having a centroid-to-centroid distance of 4.575 Å and C18-H18... $\pi$  distance of 3.282 Å, as shown in figure 4.

### Supplementary material

Crystallographic data, tables of atomic coordinates and thermal parameters, and full lists of bond lengths and angles have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 298114 for (*Nca<sub>2</sub>en*) and 616478 for (*Complex*). Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44 1223 336-033; Email: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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