# The Crystal and Molecular Structures of Two Isonicotinato N-Oxide Complexes of Cu(II)

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The crystal and molecular structures of diaquabis(isonicotinato N-oxide)copper(II), [Cu(N-inicO)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] (I) and diaquabis(isonicotinato N-oxide)bis(pyridine)copper(II), [Cu(N-inicO)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>(py)<sub>2</sub>] (I) have been determined by X-ray diffraction and refined to final I-values of 0.047 and 0.040 for I and I-respectively. The crystals of I- are monoclinic. The space group is I-c2/c, with I-d and a cell of dimensions I-and I-and I-and I-and I-c10.768(3) I-and I

space group is  $P\bar{1}$ , with Z=1 and a cell of dimensions a=7.036(5), b=9.640(3), c=9.752(4) Å,  $\alpha=110.05(3)$ ,  $\beta=70.95(5)$  and  $\gamma=104.28(5)^{\circ}$ .

Both molecules are centrosymmetric. The copper ion in 1 is surrounded by four oxygen atoms in a square-planar arrangement with Cu-O bond lengths of 1.934(4) Å from the carboxylato ligands and 1.930(2) Å from the aqua ligands, and by two out-of-plane carboxylato oxygen atoms with very long Cu-O distances of 2.691(4) Å. The coordination geometry around the copper

Table 1. Details of the data collection.

Compound	$[Cu(N-inicO)_2(H_2O)_2]$	$[Cu(N-inicO)_2(H_2O)_2(py)_2]$	
Crystal data			
Space group Unit cell	C2/c	ΡĪ	
a (Å) b c	14.136(7) 9.791(3) 10.768(3)	7.036(5) 9.640(3) 9.752(4)	
α(°) β	90.0 110.91(3) 90.00	110.05(3) 70.95(5) 104.28(5)	
$Z^{\stackrel{\gamma}{V}(\mathring{ m A}^3)}$	1392.3(9) 4	580.6(Š) 1	
$\mu$ Mo $K\alpha$ (cm <sup>-1</sup> ) M.W.	16.8 375.78	10.2 533.98	
Intensity measurements			
Collection Radiation Scan range Scan rate ° min <sup>-1</sup> Refl. meas. obs. $(I>3\sigma(I))$ $R(=\Sigma  F_o - F_c  /\Sigma F_o )$	$\theta/2\theta$ $MoKa$ $4^{\circ} < 2\theta < 65^{\circ}$ $1.5$ to $20.0$ $2924$ $1477$ $0.047$	θ/2θ Mo <i>Kα</i> 4°<2θ<50° 1.5 to 20.0 2338 1886 0.040	

ion in 2 can be described as an elongated octahedron with equatorial Cu-O and Cu-N bonds of 1.973(1) and 2.006(1) Å, respectively, and with axial Cu-O aqua bonds of 2.509(1) Å.

The isonicotinato N-oxide group coordinates to the copper ion through both carboxylato oxygen atoms in I and through one carboxylato oxygen atom for 2. There is no coordination through the N-oxide oxygen atom.

Of 3d metal compounds containing isonicotinic acid N-oxide, the structures of Mn(II), Fe(II), Co(II) and Ni(II) compounds 1,2 and tetranuclear

tetraaqua- $\mu_3$ -hydroxo- $\mu$ -sulfatotetrakis- $\mu$ -(isonicotinato N-oxide)tetracopper(II) have been described earlier. The isonicotinato N-oxide group coordinates to the central metal ion through only one carboxylato oxygen atom in the Mn(II) complex and through both carboxylato oxygen atoms to adjacent Cu atoms in the tetranuclear Cu(II) complex. Coordination to the central metal ion does not occur through the N-oxide oxygen atom. The Fe(II), Co(II) and Ni(II) compounds of isonicotinic acid N-oxide are ionic and their crystals form an isomorphous series. Hexaaqua metal(II) cations and isonicotinate

Table 2. Final positional parameters for non-hydrogen atoms with e.s.d's in parentheses and  $U_{eq}$  ( $^{A}\times 10^{3}$ ).

Atom	x	у	z	$U_{ m eq}^{\;\;a}$
[Cu(N-inicO	$(H_2O)_2$			
Cu(1)	0.7500	0.2500	0.5000	25.9(3)
O(1)	0.5553(2)	0.3227(3)	0.1177(3)	40(2)
O(2)	0.6849(3)	0.0922(3)	-0.3420(3)	59(2)
O(3)	0.6923(2)	0.3166(3)	-0.3735(3)	39(2)
O(4)	0.6171(2)	0.2284(4)	0.3659(2)	48(2)
N(1)	0.5850(2)	0.2945(3)	0.0157(3)	27(1)
C(1)	0.5980(4)	0.1618(5)	-0.0156(4)	44(2)
C(2)	0.6260(4)	0.1337(4)	-0.1221(4)	42(2)
C(3)	0.6427(3)	0.2418(5)	-0.1967(3)	27(2)
C(4)	0.6305(4)	0.3770(5)	-0.1615(4)	39(2)
C(5)	0.6012(4)	0.4012(4)	-0.0542(4)	38(2)
C(6)	0.6750(3)	0.2115(4)	-0.3132(4)	32(2)
[Cu(N-inicO	$O_{2}(H_{2}O)_{2}(py)_{2}$			
Cu(1)	0.0000	0.0000	0.0000	30.80(7)
O(1)	0.2483(1)	0.4039(1)	0.3201(1)	58.8(4)
O(2)	0.0539(1)	0.8406(1)	0.0599(1)	41.0(3)
O(3)	0.7375(1)	0.7281(1)	0.1401(1)	46.1(4)
O(4)	0.3167(1)	0.1583(1)	0.0660(1)	48.9(4)
N(1)	0.1707(1)	0.4822(1)	0.2707(1)	38.4(4)
N(2)	0.8254(1)	0.0818(1)	0.2089(1)	33.1(4)
C(1)	0.2959(1)	0.5650(1)	0.1793(1)	39.3(5)
C(2)	0.2182(1)	0.6511(1)	0.1304(1)	35.7(5)
C(3)	0.0114(1)	0.6518(1)	0.1712(1)	29.7(4)
C(4)	0.8859(1)	0.5635(1)	0.2615(1)	37.7(5)
C(5)	0.9686(2)	0.4813(1)	0.3121(1)	41.8(5)
C(6)	0.9226(1)	0.7466(1)	0.1195(1)	31.3(4)
C(7)	0.6471(1)	0.1236(1)	0.2286(1)	38.6(5)
C(8)	0.5236(2)	0.1789(1)	0.3702(1)	46.2(6)
C(9)	0.5803(2)	0.1897(1)	0.4963(1)	44.9(5)
C(10)	0.7625(2)	0.1468(1)	0.4765(1)	45.9(5)
C(11)	0.8802(1)	0.0941(1)	0.3319(1)	39.0(5)

 $<sup>^{</sup>a}U_{eq}=\frac{1}{3}(U_{11}+U_{22}+U_{33}).$ 

N-oxide anions are found in the crystal lattices.

This experiment sought to determine the structures of diaquabis(isonicotinato N-oxide)Cu(II), [Cu(N-inicO)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] and diaquabis(isonicotinato N-oxide)bispyridinecopper(II), [Cu(N-inicO)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>(py)<sub>2</sub>]. The spectral and magnetic properties of the former have been studied earlier by Gelfand *et al.*<sup>4</sup> They described [Cu(N-ionicO)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] as a tetracoordinated polymeric complex, where isonicotinato groups form single bridges between copper(II) ions through N-O oxygen and COO oxygen, the water molecules being exclusively lattice water. Magnetic susceptibility  $\chi_g$  at 295 K is  $1534 \times 10^6$  cgsu and the magnetic moment  $\mu_{\rm eff}$  is 1.91 B.M.<sup>4</sup>

### **EXPERIMENTAL**

Preparation of the compounds. The royal blue crystals of  $[Cu(N-inicO)_2(H_2O)_2]$  (1) were prepared as reported earlier.<sup>1,3</sup>

Recrystallization of 1 from ethanolic pyridine solution gave beautiful blue crystals of an unstable pyridine derivative,  $[Cu(N-inicO)_2(H_2O)_2-(py)_2]$  (2).

Table 3. Final positional parameters and isotropic thermal parameters ( $\times 10^2$ ) for hydrogen atoms and their standard deviations.

	x	у	z	U		
[Cu(N-inicO) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]						
H(1) H(2) H(3) H(4) H(5) H(6)	0.113(5) 0.641(5) 0.358(5) 0.594(4) 0.626(5) 0.099(4)	0.542(7) 0.992(7) 0.517(6) 0.493(6) 0.255(7) 0.775(6)	0.075(7) 0.354(7) 0.214(6) 0.473(5) 0.313(6) 0.393(5)	13(3) 10(2) 7(2) 6(2) 8(2) 8(2)		
[Cu( <i>N</i> -inicO) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> (py <sub>2</sub> )]						
H(1) H(2) H(3) H(4) H(5) H(6) H(7) H(8) H(9) H(10) H(11)	0.445(2) 0.321(2) 0.723(1) 0.881(2) 0.601(2) 0.395(2) 0.504(2) 0.188(1) 0.029(1) 0.331(2) 0.286(2)	0.571(1) 0.707(1) 0.554(1) 0.420(1) 0.098(1) 0.210(1) 0.779(1) 0.842(1) 0.065(1) 0.175(1) 0.219(1)	0.165(1) 0.064(1) 0.297(1) 0.382(1) 0.130(1) 0.388(1) 0.411(1) 0.306(1) -0.004(1) 0.143(1)	4.3(3) 5.5(3) 4.1(3) 5.4(3) 5.7(3) 8.0(4) 6.6(3) 4.8(3) 4.6(3) 6.9(3) 7.2(4)		

Copper was analyzed by standard EDTA titrations. The chemical analysis for C, N and H was performed by the Pulp and Paper Research Institute, Espoo, Finland.

Analytical results for [Cu(*N*-inicO)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>]. Calculated: Cu 16.91 %, C 38.36 %, N 7.45 %,

Table 4. Selected interatomic distances (Å) and angles (°) and their standard deviations for [Cu(N-inicO)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>].

Environment of copper	
Cu(1)-O(2)	2.691(4)
Cu(1)-O(3)	1.934(4)
Cu(1)-O(4)	1.930(2)
O(2)-Cu(1)-O(3)	54.7(1)
O(2)-Cu(1)-O(4)	92.9(1)
O(3) - Cu(1) - O(2)	125.2(1)
O(3)-Cu(1)-O(4) O(4)-Cu(1)-O(2)	88.8(1) 87.1(1)
O(4)-Cu(1)-O(2) O(4)-Cu(1)-O(3)	91.2(1)
	)
Pyridine N-oxide group	4.000(5)
N(1)-O(1)	1.337(5)
N(1)-C(1) N(1)-C(5)	1.371(6) 1.355(6)
C(1)-C(2)	1.369(8)
C(2)-C(3)	1.398(7)
C(3)-C(4)	1.404(7)
C(4)-C(5)	1.379(7)
O(1)-N(1)-C(1)	120.4(4)
O(1)-N(1)-C(5) C(1)-N(1)-C(5)	117.5(4)
	122.0(4)
N(1)-C(1)-C(2) C(1)-C(2)-C(3)	120.0(4) 119.2(4)
C(1)-C(2)-C(3) C(2)-C(3)-C(4)	119.7(4)
C(3) - C(4) - C(5)	119.4(4)
Carboxylato group	( )
	1 500(6)
C(6)-C(3) C(6)-O(2)	1.509(6) 1.229(6)
C(6) - O(3)	1.286(5)
, , , , ,	` ,
O(2)-C(6)-O(3)	125.0(4)
O(2)-C(6)-C(3) O(3)-C(6)-C(3)	119.5(4) 115.5(4)
0(3) 0(0) 0(3)	113.3(1)
H(1)-C(1)	1.49(7)
H(2) – C(2)	1.29(7)
H(3)-C(4) H(4)-C(5)	1.22(6) 1.09(6)
H(4)-C(5) H(5)-O(4)	0.68(7)
H(6)-O(4)	0.64(6)

Table 5. Selected interatomic distances (Å) and angles (°) and their standard deviations for [Cu(N-inicO)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>(py)<sub>2</sub>].

Environment of copper	
Cu(1)-N(2)	2.006(1)
Cu(1) – O(2)	1.973(1)
Cu(1)-O(4)	2.509(1)
N(2)-Cu(1)-O(2) N(2)-Cu(1)-O(4)	88.63(3) 92.39(2)
O(2)-Cu(1)-N(2)	91.37(3)
O(2)-Cu(1)-O(4)	91.34(3)
O(4)-Cu(1)-O(2) O(4)-Cu(1)-N(4)	88.66(3) 87.61(2)
	(-)
Pyridine N-oxide group	
N(1)-O(1) N(1)-C(1)	1.314(1) 1.355(1)
N(1) - C(1) N(1) - C(5)	1.334(1)
C(1)-C(2)	1.371(2)
C(2)-C(3) C(3)-C(4)	1.379(1)
C(3)-C(4) C(4)-C(5)	1.385(1) 1.376(2)
	119.52(8)
O(1)-N(1)-C(1) O(1)-N(1)-C(5)	120.07(8)
C(1)-N(1)-C(5)	120.41(10) 120.33(9)
N(1)-C(1)-C(2) C(1)-C(2)-C(3)	120.33(8)
C(1) $C(2)$ $C(3)C(2)$ $C(3)$ $C(4)C(5)$ $C(4)$ $C(5)$	118.33(10)
C(5)-C(4)-C(3) C(4)-C(5)-N(1)	120.02(9) 120.53(9)
C(4) C(3) IV(1)	120.55(9)
Carboxylato group	
C(6)-C(3) C(6)-O(2)	1.510(1)
C(6)-C(2) C(6)-C(3)	1.269(1) 1.228(1)
O(2)-C(6)-O(3)	127.06(10)
O(2)-C(6)-C(3) O(3)-C(6)-C(3)	114.05(8)
O(3)-C(6)-C(3)	118.87(8)
Pyridine ring	
N(2)-C(7)	1.346(1)
N(2)-C(11)	1.336(1)
C(7)-C(8) C(8)-C(9)	1.374(1) 1.377(2)
C(8)-C(9) C(9)-C(10)	1.377(2)
C(10)-C(11)	1.373(1)
C(7)-N(2)-C(11)	118.18(7)
N(2)-C(7)-C(8) C(7)-C(8)-C(9)	121.81(10) 119.62(11)
C(8)-C(9)-C(10)	118.65(8)
C(9)-C(10)-C(11) N(2)-C(11)-C(10)	118.94(11)
N(2)-C(11)-C(10)	122.79(10)

O 34.06 %, H 3.22 %. Found: Cu 16.43 %, C 38.66 %, N 7.18 %, H 3.16 %.

Data collection. A Syntex  $P2_1$ , four-circle diffractometer employing graphite monochromatized Mo $K\alpha$ -radiation was used to measure the crystal and intensity data of both compounds. The unit cell parameters were calculated by least squares refinement of 15 high order reflections.

After centering, the crystal of 2 was coated with glue to prevent decomposition in air by loss of pyridine.

Details of the data collections, including the crystal data, may be seen in Table 1. The data were corrected for Lorentz and polarization factors but not for absorption.

Structure determination. The structure of *I* was solved by direct methods<sup>5</sup> while for 2 the locations of the copper atom and three oxygen atoms were determined from a Patterson map. The refinement of the structures using unit weights was carried out by the X-Ray System programs.<sup>6</sup> All hydrogen atoms were located using difference Fourier syntheses. The final *R*-values were 0.047 and 0.040 for *I* and 2, respectively. A refinement in space group *Cc* for *I* was also tried but it did not bring about any significant improvements. The scattering factors for Cu, C, N and O were those of Cromer and Mann<sup>7</sup> and for hydrogen atoms those reported by Stewart *et al.*<sup>8</sup>

Tables 2 and 3 present the final atomic coordinates and the thermal parameters with their standard deviations for both compounds. Lists of structure factors and anisotropic thermal parameters are available from the authors upon request.

## DESCRIPTION AND DISCUSSION OF THE STRUCTURES

The labeling and the structure of the molecules are given in Fig. 1 for  $[Cu(N-inicO)_2(H_2O)_2]$  and in Fig. 2 for  $[Cu(N-inicO)_2(H_2O)_2(py)_2]$ . Bond lengths and angles are listed in Tables 4 and 5.

Both copper(II) complexes are monomeric containing two chelating isonicotinato *N*-oxide and two aqua ligands per molecule for [Cu(*N*-inicO)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] and two monodentate isonicotinato *N*-oxide, two aqua and two pyridine ligands per molecule for [Cu(*N*-inicO)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>(py)<sub>2</sub>]. The molecules are joined in the crystal lattice by

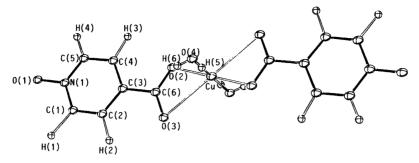


Fig 1. The labeling and structure of  $[Cu(N-inicO)_2(H_2O)_2]$ . The ellipsoids are not scaled with thermal motion.

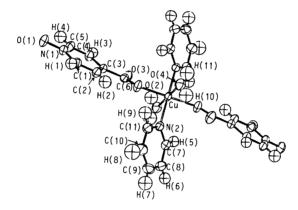


Fig. 2. The labeling and structure of  $[Cu(N-inicO)_2(H_2O)_2(py)_2]$ . Thermal ellipsoids are drawn to enclose the 50 % probability level.

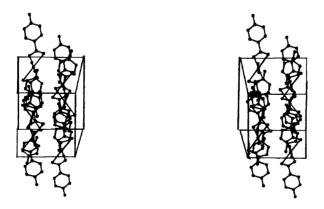


Fig. 3. Stereoview of the packing of  $[Cu(N-inicO)_2(H_2O)_2]$  within the unit cell. Hydrogen atoms have been excluded for clarity.

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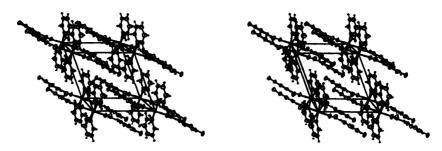


Fig. 4. Stereoview of the structure, illustrating the packing of  $[Cu(N-inicO)_2(H_2O)_2(py)_2]$  within the unit cell.

hydrogen bonds, Table 6. The packing in the crystals is shown in Figs. 3 and 4.

In the  $[Cu(N-inicO)_2(H_2O)_2]$  complex, oxygen atoms surround the copper ion forming a square planar arrangement. In addition there are two "out-of-plane" carboxylato oxygen atoms with very long Cu(1)-O(2) distances of 2.691(4) Å. oxygen-copper The carboxylato bond. Cu(1) - O(3)1.934(4) and the agua oxygen-copper bond, Cu(1)-O(4) 1.930(2) Å are equal. Angles O(3)-Cu(1)-O(4) and O(2)-Cu(1)-O(3) are 91.2(1) and 54.8(1)°, respectively. The angle O(2)-C(6)-O(3) in the carboxylato group is 125.0(4)°. The atoms C(3), C(6), O(2) and O(3) form a plane from which the copper atom is offset by 0.245 Å (Table 7).

The angle between the planes of the carboxylato group and pyridine ring is 1.7°. If the normal to the Cu(1), O(3), O(4) plane is considered as the "tetragonal" axis, then the angle between the "out-of-plane" Cu(1)-O(2) direction and the "tetragonal" axis is 35.5°.

The coordination geometry around the copper atom can be regarded as a square plane or a tetragonally distorted octahedron, depending upon whether or not the "out-of-plane" oxygen atoms are considered bonded to the Cu ion. However, we prefer to describe the coordination as a six-coordination with an extremely tetragonally distorted octahedral configuration although the long Cu(1)-O(2) distance indicates very weak interaction. Structures of copper(II) complexes have been presented where the geometry and coordination of the carboxylato group are similar to the coordination of the carboxylato group in  $[Cu(N-inicO)_2(H_2O)_2]^{.9-12}$  The lengths of the equatorial Cu-O bonds, the "out-ofplane" Cu-O bond, 2.691(4) Å and the angle of O(2)-Cu(1)-O(3) in  $[Cu(N-inicO)_2(H_2O)_2]$ , have values close to the corresponding values trans-bis(chloroacetato)-bis(2-picoline)copper(II)  $(1.975(5), 2.707(7) \text{ Å}, 53.9^{\circ})$ , in diaquabis(hydrogen o-phtalato)copper(II)  $(1.930(8), 1.967(8), 2.677(5) \text{ Å}, 54.4(2)^{\circ})$ , 11 and

Table 6. Hydrogen bonds.

	A−H···B	A-H	$H \cdots B$	A-B	∠AHB(°)
$[Cu(N-inicO)_2(H_2O)_2]$	$O(4)-H(5)\cdots O(1)$	0.68(7)	2.09(6)	2.663(4)	143(6)
	$O(4)^{a}-H(6)\cdots O(1)^{b}$	0.64(6)	2.19(6)	2.671(5)	133(6)
$[\mathrm{Cu}(N\text{-inicO})_2(\mathrm{H}_\mathrm{O})_2(\mathrm{py})_2]$	$O(4)-H(10)\cdots O(3)^{c}$	0.72(1)	2.09(1)	2.757(1)	154(1)
	$O(4)-H(11)\cdots O(1)$	0.78(1)	2.01(1)	2.782(1)	168(1)
Symmetry code a: $x-\frac{1}{2}$ , b: $-x+\frac{1}{2}$ c: $-x+\frac{1}{2}$	$y + \frac{1}{2}, z$ $\frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$ $\frac{1}{2}, -y + 1, -z$				

Table 7. Deviations (Å) of atoms from least-squares planes.

[Cu(N-inicO) <sub>2</sub> (H	$I_2O)_2$					
Plane 1: O(2), O(3), C(6), C(3)						
O(2) 0.003	O(3) 0.003	C(6) -0.008	C(3) 0.002	Cu(1) 0.245		
Plane 2: N(1), N(1) -0.006	C(1), C(2), C(3) C(1) 0.007		C(3) -0.003	C(4) 0.004 C(5	) 0.000	
$[Cu(N-inicO)_2(H_2O)_2(py)_2]$ Plane 3: O(2), O(3), C(6), C(3)						
O(2) $0.001$		C(6) -0.034	C(3) -0.010	Cu(1) 0.247		

in bis(chloroacetato)-(N,N,N',N'-tetramethylethylenediamine)copper(II) (1.975-1.977(3), 2.624-2.627(3) Å,  $55.6-55.7(1)^{\circ}$ ). However, the copper atom in bis(dichloroacetato)bis(2-picoline)copper(II) has been considered pentacoordinated since only one of the dichloroacetato groups coordinates through both carboxylato oxygen atoms. <sup>10</sup>

The structure of the pyridine derivative seems to favour an explanation based on hexacoordinadiaquabis(isonicotinato When tion, too. N-oxide)copper(II) was crystallized from ethanolic pyridine solution, two pyridine molecules joined the coordination sphere of the copper atom so that both the long bond between carboxylato oxygen and copper disappeared and the bond between the agua oxygen atom and the copper atom increased in length from 1.930(2) to 2.509(1) Å. The geometry around the copper atom is a distorted elongated octahedron with basal Cu-O bonds of 1.973(1) Å and Cu-N bonds of 2.001(1) Å. In both Cu(II) complexes under study the isonicotinato N-oxide ligand coordinates only through carboxylato oxygen atoms, being either bidentate or monodentate.

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