

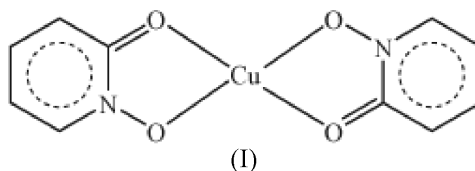
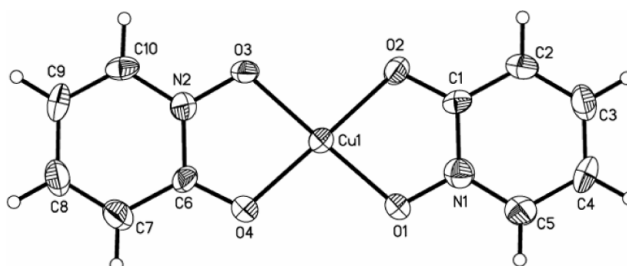
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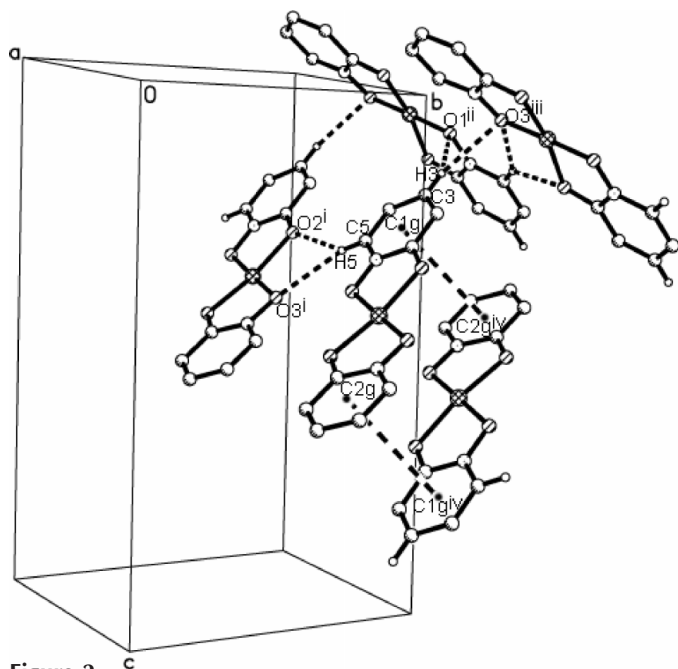
## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å  
 $R$  factor = 0.044  
 $wR$  factor = 0.113  
Data-to-parameter ratio = 11.8For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Bis(2-oxopyridinato *N*-oxide- $\kappa^2\text{O},\text{O}'$ )copper(II)The title complex,  $[\text{Cu}(\text{C}_5\text{H}_4\text{NO}_2)_2]$ , displays a *trans*- $\text{O}_2(\text{N}$ -oxide) $\text{O}_2$  square-planar coordination geometry for the  $\text{Cu}^{\text{II}}$  centre. In the crystal structure, the molecules assemble into a three-dimensional structure by way of intermolecular  $\pi$ - $\pi$  interactions and weak intermolecular  $\text{C}-\text{H}\cdots\text{O}$  interactions.Received 4 January 2005  
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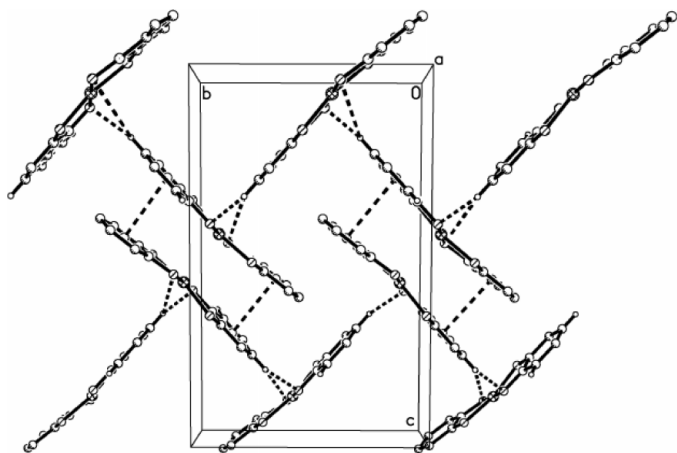
## Comment

Many metal complexes with pyridine *N*-oxide and its derivatives have been reported in the past few decades as a result of their interesting coordination chemistry (Karayannis *et al.*, 1973, 1976; Landers & Phillips, 1982; Casellato *et al.*, 1983; Riley *et al.*, 1983; Scarrow *et al.*, 1985; Lobana & Singh, 1995). However, metal complexes with chelating derivatives of pyridine *N*-oxide with donor atoms in the 2-position, such as 2-hydroxypyridine *N*-oxide (opo) (Hubbard *et al.*, 1979; Landers & Phillips, 1981), have been less well studied and little work has been carried out on the X-ray crystal structures of complexes of this class (Casellato *et al.*, 1983; Riley *et al.*, 1983). Here, we report the synthesis and structure of the square-planar title compound,  $[\text{Cu}(\text{opo})_2]$ , (I). A previous study of  $[\text{Cu}(\text{opo})_2]$  suggested that it was a mononuclear five- or six-coordinate complex (Landers & Phillips, 1981).Compound (I) has a square-planar geometry around the central Cu atom, which is coordinated by two opo ligands (Fig. 1 and Table 1). The two *N*-oxide O atoms are in a *trans* configuration. The  $\text{CuO}_4$  moiety has approximate local  $\bar{1}$  symmetry. The whole molecule is nearly planar, with a maximum deviation from the mean plane of 0.284 (3) Å for atom O3. The average Cu—O bond distance of 1.919 (3) Å is

**Figure 1**  
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.



**Figure 2**  
Detail of the structure of (I), showing the intermolecular  $\pi$ - $\pi$  and weak intermolecular C-H $\cdots$ O interactions (dashed lines). C1<sup>g</sup> and C2<sup>g</sup> represent the centroids of the pyridine rings (see text). All H atoms, except H3 and H5, have been omitted for clarity. [Symmetry codes: (i)  $1 + x, y, z$ ; (ii)  $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (iii)  $-x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (iv)  $-x, 2 - y, 1 - z$ ].



**Figure 3**  
The three-dimensional packing structure of (I). Dashed lines indicate  $\pi$ - $\pi$  stacking and C-H $\cdots$ O interactions. All H atoms, except H3 and H5, have been omitted for clarity.

comparable with those found in other complexes with a CuO<sub>4</sub> core, e.g. bis(1,2-dimethyl-3-hydroxypyridin-4-one-*O,O'*)copper(II) [1.920 (2) Å; El-Jammal *et al.*, 1994] and bis(1,2-diethyl-3-hydroxypyridin-4-one-*O,O'*)copper(II) [1.918 (2) Å; El-Jammal *et al.*, 1994].

There are two intermolecular  $\pi$ - $\pi$  interactions and four weak intermolecular C-H $\cdots$ O interactions in the crystal structure of (I) (Fig. 2 and Table 2). Chains of molecules are formed along the *a* axis through two weak intermolecular interactions between neighbouring molecules [C5-H5 $\cdots$ O2<sup>i</sup> and C5-H5 $\cdots$ O3<sup>i</sup>; symmetry code: (i)  $1 + x, y, z$ ]. Neigh-

bouring chains of molecules are connected to each other through two further weak interactions, resulting in a layer structure [C3-H3 $\cdots$ O1<sup>ii</sup> and C3-H3 $\cdots$ O3<sup>iii</sup>; symmetry codes: (ii)  $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (iii)  $-x, \frac{1}{2} + y, \frac{1}{2} - z$ ; Fig. 3].

Neighbouring layers interact further with each other through  $\pi$ - $\pi$  stacking interactions between the ring systems of neighbouring pyridine rings, with Cg1 $\cdots$ Cg2<sup>iv</sup> 3.828 (2) Å and Cg2 $\cdots$ Cg1<sup>iv</sup> 3.828 (2) Å, where Cg1 and Cg2 are the centroids of the pyridine rings containing atoms C1 and C6, respectively [symmetry code: (iv)  $-x, 2 - y, 1 - z$ ].

## Experimental

To a methanol solution (25 ml) of 2-hydroxypyridine *N*-oxide (0.55 g, 5.0 mmol) and NaOCH<sub>3</sub> (0.27 g, 5.0 mmol) was added CuCl<sub>2</sub>·4H<sub>2</sub>O (0.43 g, 2.5 mmol) with stirring. The resulting solution was stirred at room temperature for 4 h and then filtered. Blue block crystals of (I) suitable for X-ray structure analysis were obtained by recrystallizing the blue precipitate from dimethylformamide (60% yield). Analysis calculated for C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>O<sub>4</sub>Cu: C 42.33, H 2.84, N 9.87%; found: C 42.50, H 2.86, N 10.02%. Spectroscopic analysis: IR (KBr,  $\nu$ , cm<sup>-1</sup>): 3094 (*m*), 1623 (*s*, C-O), 1548 (*s*), 1503 (*s*), 1443 (*m*), 1361 (*s*), 1232 (*m*), 1183 (*s*, N-O), 1142 (*w*), 1109 (*w*), 1027 (*w*), 947 (*w*), 886 (*m*), 787 (*s*), 754 (*s*), 634 (*s*), 545 (*w*), 452 (*w*).

### Crystal data

[Cu(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>O<sub>4</sub>)<sub>2</sub>]  
M<sub>r</sub> = 567.45  
Monoclinic, *P*2<sub>1</sub>/*c*  
*a* = 6.873 (5) Å  
*b* = 9.721 (5) Å  
*c* = 15.523 (2) Å  
 $\beta$  = 96.49 (1)°  
*V* = 1030.5 (9) Å<sup>3</sup>  
*Z* = 2

*D<sub>x</sub>* = 1.829 Mg m<sup>-3</sup>  
Mo K $\alpha$  radiation  
Cell parameters from 25 reflections  
 $\theta$  = 3.0–25.0°  
 $\mu$  = 2.12 mm<sup>-1</sup>  
*T* = 293 (2) K  
Block, blue  
0.30 × 0.20 × 0.15 mm

### Data collection

Siemens P4 diffractometer  
 $\omega/2\theta$  scans  
Absorption correction:  $\psi$  scan  
(*XPREP*; Bruker, 2000)  
*T*<sub>min</sub> = 0.612, *T*<sub>max</sub> = 0.734  
4108 measured reflections  
1820 independent reflections  
1595 reflections with *I* > 2 $\sigma$ (*I*)

*R*<sub>int</sub> = 0.029  
 $\theta$ <sub>max</sub> = 25.0°  
*h* = -8 → 8  
*k* = -11 → 7  
*l* = -15 → 18  
3 standard reflections  
every 97 reflections  
intensity decay: 0.2%

### Refinement

Refinement on *F*<sup>2</sup>  
 $R[F^2 > 2\sigma(F^2)] = 0.044$   
 $wR(F^2) = 0.113$   
*S* = 0.99  
1820 reflections  
154 parameters  
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 1.66P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.33 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.70 \text{ e } \text{Å}^{-3}$

**Table 1**

Selected geometric parameters (Å, °).

|           |             |           |             |
|-----------|-------------|-----------|-------------|
| Cu1—O1    | 1.891 (3)   | Cu1—O3    | 1.918 (3)   |
| Cu1—O4    | 1.916 (3)   | Cu1—O2    | 1.951 (3)   |
| O1—Cu1—O4 | 97.10 (12)  | O1—Cu1—O2 | 84.02 (12)  |
| O1—Cu1—O3 | 178.28 (12) | O4—Cu1—O2 | 169.56 (14) |
| O4—Cu1—O3 | 83.99 (12)  | O3—Cu1—O2 | 94.67 (12)  |

**Table 2**  
Hydrogen-bonding geometry (Å, °).

| $D-H\cdots A$             | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------|-------|-------------|-------------|---------------|
| C3–H3...O3 <sup>iii</sup> | 0.93  | 2.57        | 3.332 (5)   | 140           |
| C3–H3...O1 <sup>ii</sup>  | 0.93  | 2.58        | 3.343 (5)   | 139           |
| C5–H5...O3 <sup>i</sup>   | 0.93  | 2.55        | 3.394 (5)   | 150           |
| C5–H5...O2 <sup>i</sup>   | 0.93  | 2.64        | 3.428 (6)   | 143           |

Symmetry codes: (i)  $1+x, y, z$ ; (ii)  $1-x, \frac{1}{2}+y, \frac{1}{2}-z$ ; (iii)  $-x, \frac{1}{2}+y, \frac{1}{2}-z$ .

All H atoms were positioned geometrically and refined as riding, with C–H distances of 0.93 Å and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: *XSCANS* (Bruker, 2000); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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