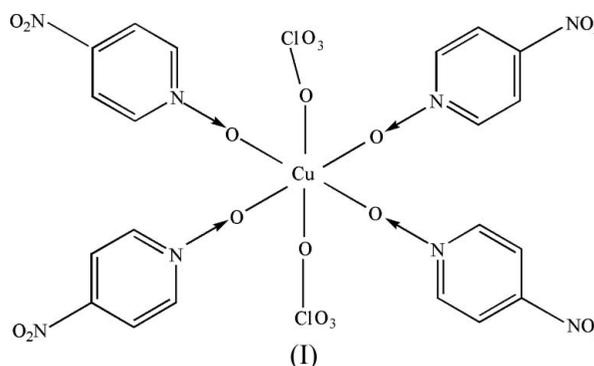


Jing-Min Shi,* Ju-Na Chen,
Chang-Ju Wu and
Lian-Dong LiuDepartment of Chemistry, Shandong Normal
University, Jinan 250014, People's Republic
of ChinaCorrespondence e-mail:
shijingmin@beelink.com

Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.043
 wR factor = 0.130
Data-to-parameter ratio = 11.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Tetrakis(4-nitropyridine *N*-oxide- κ O)-
bis(perchlorato)copper(II)In the title mononuclear complex, $[\text{Cu}(\text{ClO}_4)_2(\text{C}_5\text{H}_4\text{N}_2\text{O}_3)_4]$, the six-coordinated Cu^{II} ion lies on an inversion centre and presents a slightly distorted octahedral geometry. A π - π stacking interaction is observed between symmetry-related pyridine rings.Received 4 October 2005
Accepted 9 November 2005
Online 16 November 2005

Comment

Pyridine *N*-oxide and its derivatives are useful ligands, and a number of complexes have been synthesized with these compounds as bridging ligands and terminal ligands (Watson, 1969; Shi *et al.*, 2005). These complexes are interesting for understanding the relationship between the coordination modes and the respective metal ions. We report here the synthesis and structure of such a Cu^{II} complex, (I).

The structure of complex (I) is shown in Fig. 1. The Cu^{II} ion is located on an inversion centre and displays a slightly distorted $[\text{CuO}_6]$ octahedral coordination geometry (Table 1). The four coordinated O atoms belonging to four 4-nitropyridine *N*-oxide molecules are located in a square plane. The octahedral geometry is completed by two O atoms from two perchlorate ions, in axial positions. Obviously, (I) exhibits a marked Jahn–Teller distortion. The IR spectrum shows strong absorptions at 1145 , 1122 and 1087 cm^{-1} , which are attributed to the vibrations of the perchlorate ions, consistent with their involvement in coordination (Wickenden & Krause, 1965). The angles between the CuO_4 square plane and pyridine planes are $81.17(14)$ ($\text{N}2/\text{C}1-\text{C}5$ plane) and $76.14(14)^\circ$ ($\text{N}3/\text{C}6-\text{C}10$ plane), yielding a four-blade propeller geometry for the complex (Fig. 1).

There is a significant π - π stacking interaction between adjacent pyridine rings; the relevant distances are $\text{Cg}1 \cdots \text{Cg}1^{\text{ii}} = 3.848(2)\text{ \AA}$ and $\text{Cg}1 \cdots \text{Cg}1^{\text{ii}}_{\text{perp}} = 3.507\text{ \AA}$ [symmetry code: (ii) $1 - x, 2 - y, 1 - z$; $\text{Cg}1$ is the centroid of the $\text{N}3/\text{C}6-\text{C}10$ pyridine ring; $\text{Cg}1 \cdots \text{Cg}1^{\text{ii}}_{\text{perp}}$ is the perpendicular distance from $\text{Cg}1$ to $\text{Cg}1^{\text{ii}}$].

Experimental

4-Nitropyridine *N*-oxide (0.1074 g, 0.767 mmol, in H₂O (10 ml) was added to an aqueous solution (10 ml) containing Cu(ClO₄)₂·6H₂O (0.0802 g, 0.216 mmol), and the solution was stirred for a few minutes. Red single crystals were obtained after the solution was allowed to stand at 298 K for three weeks.

Crystal data

[Cu(ClO₄)₂(C₅H₄N₂O₃)₄]
M_r = 822.85
 Monoclinic, *P*2₁/*c*
a = 9.9557 (19) Å
b = 9.8964 (19) Å
c = 15.715 (3) Å
 β = 101.938 (2)°
V = 1514.9 (5) Å³
Z = 2

D_x = 1.804 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 5055 reflections
 θ = 2.5–27.9°
 μ = 1.00 mm⁻¹
T = 298 (2) K
 Prism, red
 0.48 × 0.26 × 0.21 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.645, *T_{max}* = 0.817
 7546 measured reflections

2668 independent reflections
 2351 reflections with *I* > 2σ(*I*)
R_{int} = 0.035
 θ_{max} = 25.0°
h = -11 → 11
k = -11 → 9
l = -18 → 16

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.043
wR(*F*²) = 0.130
S = 1.02
 2668 reflections
 233 parameters
 H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0777*P*)² + 1.3498*P*]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.76 e Å⁻³
 Δρ_{min} = -0.70 e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0059 (15)

Table 1

Selected geometric parameters (Å, °).

Cu1–O3	1.908 (2)	O3–N2	1.327 (3)
Cu1–O4	1.955 (2)	O4–N3	1.334 (3)
O3–Cu1–O4 ⁱ	90.61 (9)	N2–O3–Cu1	121.73 (18)
O3–Cu1–O4	89.39 (9)	N3–O4–Cu1	116.96 (17)
O4 ⁱ –Cu1–O4	180		

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

All H atoms were placed in calculated positions and were included in the final cycles of refinement using a riding model, with C–H distances constrained to 0.93 Å and *U*_{iso}(H) = 1.2_{eq}(carrier C atom).

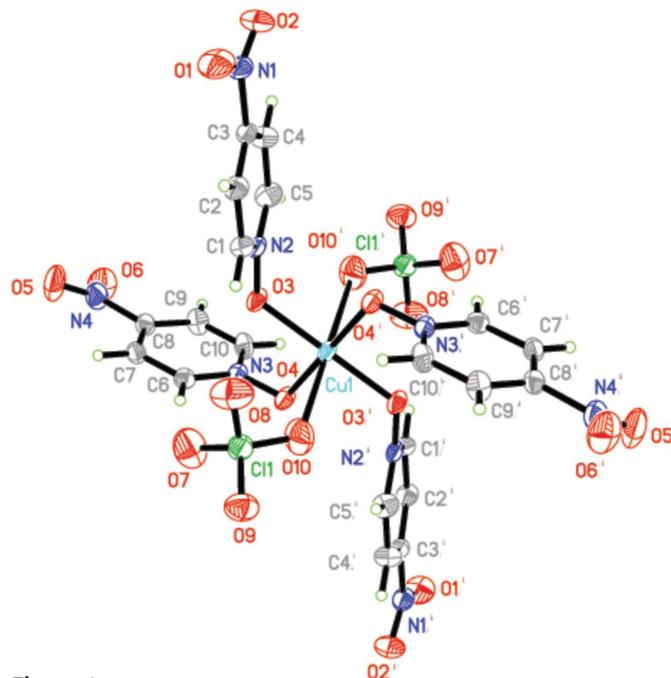


Figure 1

A view of complex (I), with the atom-numbering scheme, showing 30% probability displacement ellipsoids for non-H atoms. H atoms are shown as small spheres of arbitrary radii. (Symmetry code for primed atoms: 1 - x, 1 - y, 1 - z.)

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

The authors thank the Natural Science Foundation of China (No. 20271043) and the Natural Science Foundation of Shandong Province of China (No. Y2002B10) for support.

References

- Bruker (1997). SMART, SAINT and SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Shi, J. M., Chen, J. N. & Liu, L. D. (2005). Acta Cryst. E61, m1935–m1936.
 Watson, W. H. (1969). Inorg. Chem. 8, 1879–1886.
 Wickenden, A. E. & Krause, R. A. (1965). Inorg. Chem. 4, 404–407.