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Structure of (8-Amino-5,7-dichloroquinoline)dinitratocopper(II), $[Cu(NO_3)_2(C_9H_6Cl_2N_2)]$

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Abstract. $M_r = 400{\cdot}62$, monoclinic, $C2/c$, $a = 20{\cdot}901\ (5)$, $b = 7{\cdot}439\ (2)$, $c = 18{\cdot}708\ (4)$ Å, $\beta = 114{\cdot}59\ (3)°$, $V = 2645\ (2)$ Å^3, $Z = 8$, $D_x = 2{\cdot}01$ Mg m^{-3}, $F(000) = 1592$, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu(\text{Mo }K\alpha) = 2{\cdot}152$ mm^{-1}, room temperature, $R = 0{\cdot}016$, $R_w = 0{\cdot}021$ for 1132 observed reflections. The Cu atom displays a distorted octahedral coordination, being linked to four O atoms of two NO_3 groups and two N atoms of the aminoquinoline. The different hybridization states of the N atoms produce variations in equatorial Cu—O bond distances, while the apical Cu—O lengths are larger than the equatorial lengths: Cu—O$_{\text{eq}}$ 1·990 (2), 2·039 (3) Å; Cu—O$_{\text{ax}}$ 2·488 (3), 2·473 (3) Å.

Introduction. The title compound was synthesized in the Department of Inorganic Chemistry of the Universidad Autonoma de Barcelona, as a first step in attempts to obtain μ-NO_3 bridges between Cu^{2+} ions. Crystal structure analysis was carried out in order to elucidate the position of the nitro groups and determine the geometry of the complex.

Experimental. D_m not determined. Crystal 0·15 × 0·15 × 0·1 mm. Philips PW1100 diffractometer, monochromatized Mo $K\alpha$ radiation. Cell parameters from 25 reflections ($6 \leq \theta \leq 9°$). ω-scan technique, scan width 1°, scan speed 0·03° s^{-1}. 1265 intensities ($\theta \leq 25°$), $hkl = -24/22,\ 0/8,\ 0/22$; 1132 with $I \geq 2{\cdot}5\sigma(I)$. Three standard reflections every 2 h; no significant variation of intensity. Lp, absorption (*SHELX*76, Sheldrick, 1976) corrections, max. and min. transmission factors 0·75 and 0·64. Cu atom from Patterson synthesis, remaining atoms from weighted F_o synthesis. Full-matrix least-squares refinement (*SHELX*76); anisotropic thermal parameters, H atoms not considered; $\sum w\|F_o| - |F_c\|^2$ minimized; $w = \sigma^{-2}(F_o)$. f, f' and f'' from *International Tables for X-ray Crystallography* (1974). $(\Delta/\sigma)_{\max} = -0{\cdot}08$ [U_{11} of N(11)]. $\rho_{\max}$ and $\rho_{\min}$ on final ΔF synthesis 0·2 e Å^{-3} and $-0{\cdot}3$ e Å^{-3}. VAX-VM780 and IBM4341 computers.

Discussion. Final atomic parameters are in Table 1* and selected bond lengths and angles in Table 2. The atom numbering is shown in Fig. 1. The Cu^{2+} ion is surrounded by two N atoms of the aminoquinoline group and four O atoms of two nitro groups in a distorted octahedral shape. N(1), N(11), O(14), O(18) are in a roughly equatorial plane [deviations from the mean plane are: Cu 0·041 (3), N(1) −0·141 (4), N(11) 0·118 (4), O(14) 0·111 (4) and O(18) −0·130 (4) Å], while O(17) and O(20) occupy the axial sites [deviations of these atoms from the mean equatorial plane are −1·948 (4) and 1·743 (4) Å]. The axial

Table 1. *Atomic coordinates* (×10^4, *for* Cu ×10^5) *and equivalent isotropic thermal parameters* (Å^2)

$$B_{\text{eq}} = \tfrac{8}{3}\pi^2 \textstyle\sum U_{ij} a^*_i a^*_j \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	B_{eq}
Cu	15189 (2)	78235 (7)	21761 (3)	3·19 (8)
N(1)	660 (1)	7507 (3)	1207 (2)	2·3 (1)
C(2)	49 (2)	6862 (6)	1127 (3)	3·7 (2)
C(3)	−512 (2)	6635 (5)	413 (3)	3·5 (2)
C(4)	−461 (2)	7201 (7)	−258 (3)	3·9 (2)
C(5)	184 (2)	7953 (5)	−211 (2)	2·9 (2)
C(6)	316 (2)	8422 (5)	−844 (3)	3·5 (2)
C(7)	964 (2)	9132 (6)	−747 (3)	3·8 (2)
C(8)	1494 (2)	9194 (5)	7 (3)	2·9 (2)
C(9)	1399 (2)	8685 (6)	653 (2)	2·9 (2)
C(10)	739 (2)	7983 (5)	552 (2)	2·5 (2)
N(11)	1906 (2)	8828 (5)	1444 (2)	3·0 (1)
Cl(12)	−352 (1)	8312 (2)	−1777 (1)	5·3 (2)
Cl(13)	2294 (1)	10096 (2)	114 (1)	5·8 (2)
O(14)	1077 (1)	7003 (4)	2878 (2)	3·2 (1)
N(15)	1246 (2)	5390 (5)	2996 (2)	3·7 (1)
O(16)	1068 (2)	4494 (5)	3444 (2)	5·1 (3)
O(17)	1564 (2)	4680 (4)	2625 (2)	5·1 (3)
O(18)	2477 (1)	7751 (4)	3111 (2)	4·1 (1)
N(19)	2550 (2)	9385 (5)	3348 (2)	3·5 (1)
O(20)	2078 (1)	10392 (4)	3071 (2)	4·2 (3)
O(21)	3111 (1)	9771 (5)	3958 (2)	4·4 (3)

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* Lists of structure factors, anisotropic thermal parameters and bond lengths and angles in the ligands have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39748 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 2. *Bond distances* (Å) *and angles* (°) *for the* Cu *coordination sphere*

Cu–N(1)	1·963 (3)	Cu–O(18)	2·039 (3)
Cu–N(11)	2·002 (4)	Cu–O(20)	2·488 (3)
Cu–O(14)	1·990 (2)	Cu–O(17)	2·473 (3)
N(11)–Cu–N(1)	83·5 (1)	O(18)–Cu–O(14)	89·2 (1)
O(14)–Cu–N(1)	94·4 (1)	O(18)–Cu–O(17)	78·4 (1)
O(14)–Cu–N(11)	175·1 (1)	O(20)–Cu–N(1)	134·9 (1)
O(17)–Cu–N(1)	95·2 (1)	O(20)–Cu–N(11)	87·9 (1)
O(17)–Cu–N(11)	128·0 (1)	O(20)–Cu–O(14)	90·5 (1)
O(17)–Cu–O(14)	56·5 (1)	O(20)–Cu–O(17)	123·8 (1)
O(18)–Cu–N(1)	169·4 (1)	O(20)–Cu–O(18)	54·9 (1)
O(18)–Cu–N(11)	93·7 (1)		

Cu–O distances [mean value 2·480 (7)] are larger than the equatorial distances [mean 2·014 (24) Å]. The Cu–N(1) bond distance [1·963 (3) Å] is similar to those obtained in other compounds [weighted mean of literature values 1·96 (1) Å (Palenik, 1964; Hoy & Morris, 1967; Williams & Wallwork, 1967], while it is shorter than the Cu–N(11) bond distance [2·002 (4) Å] due to the sp^2 character of N(1); this produces a lengthening of the Cu–O bond distance opposite to N(1).

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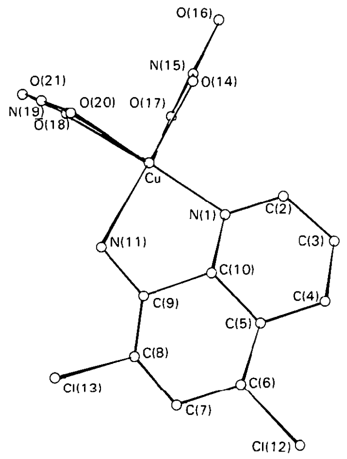

Fig. 1. A view of the complex with the numbering of the atoms.

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Triply Bridged Thiolate Complexes of Oxotungstate(V): Structures of Tetraphenylphosphonium μ-Chloro-bis(μ-phenylthiolato)-bis[dichlorooxotungstate(V)] (I), $[Ph_4P][Cl_2OW(\mu\text{-}Cl)(\mu\text{-}SC_6H_5)_2WOCl_2]$, and Tetraphenylarsonium μ-Chloro-bis(μ-p-tolylthiolato)-bis[dichlorooxotungstate(V)] (II), $[Ph_4As][Cl_2OW(\mu\text{-}Cl)\{\mu\text{-}S(p\text{-}C_6H_4CH_3)\}_2WOCl_2]$

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Abstract. (I): $M_r = 1134·71$, triclinic, $P\bar{1}$, $a = 10·8421$ (8), $b = 11·242$ (1), $c = 16·491$ (1) Å, $\alpha = 98·384$ (7), $\beta = 98·119$ (6), $\gamma = 100·267$ (7)°, $V = 1927·6$ (4) Å^3, $Z = 2$, $D_m = 1·94$ (1), $D_x = 1·955$ g cm^{-3}, $F(000) = 1084$, μ(Mo $K\alpha$) = 68·30 cm^{-1}, $\lambda = 0·71069$ Å, $T = 295$ (1) K, $R = 0·036$ for 6066 unique reflections. (II): $M_r = 1206·71$, tetragonal, $P\bar{4}$, $a = 16·173$ (1), $c = 7·8566$ (8) Å, $V = 2055·1$ (3) Å^3, $Z = 2$, $D_m = 1·94$ (1), $D_x = 1·950$ g cm^{-3}, $F(000) = 1152$, μ(Mo $K\alpha$) = 72·13 cm^{-1}, $T = 295$ (1) K, $R = 0·049$ for 1430 unique reflections. The anions consist of a greatly distorted confacial bioctahedral framework in which there is a weakly bound μ-Cl atom *trans* to two terminal O atoms. The R groups (phenyl, p-tolyl) on the bridging thiolato ligands adopt the relatively unusual axial–axial configuration.

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