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catena- μ -Perchlorato- O,O' -tris- μ -quinoxaline- N,N' -dicopper(I) Perchlorate: Structure and Infrared Spectrum

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Abstract. $[\text{Cu}_2(\text{C}_8\text{H}_6\text{N}_2)_3(\text{ClO}_4)](\text{ClO}_4)$, $M_r = 716.4$, monoclinic, $P2_1$, $a = 8.952$ (2), $b = 14.573$ (4), $c = 9.912$ (3) Å, $\beta = 94.61$ (2)°, $V = 1288.9$ (5) Å³, $D_m = 1.846$ (5), $D_x = 1.85$ g cm⁻³, $Z = 2$, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu(\text{Mo } K\alpha) = 19.3$ cm⁻¹, $F(000) = 720$, $T = 295$ K, final refinement on F [2520 data with $F_o > 6\sigma(F_o)$, $2\theta_{\text{max}} = 55.0^\circ$]: $R = 0.048$, $wR = 0.049$. The polymeric structure of the title compound consists of a three-dimensional net with all three quinoxaline molecules and one perchlorate ion forming bridges between Cu atoms. The coordination sphere of both Cu atoms is a distorted tetrahedron. The IR spectrum shows bands typical for bridging quinoxaline and perchlorate.

Introduction. Copper easily forms complexes with heterocyclic amines and the structures of many of them, especially of pyrazines, have been determined. However, only a few copper quinoxaline complexes have been discussed, but no crystal structure has been determined. Both pyrazine and quinoxaline form complexes where the ligands are bonded through one N atom to the central atom (Morosin, Hughes & Soos, 1975; Lumme, Lindroos & Lindell, 1987) or the ligands can form bridges between the metal ions (Darriet, Haddad, Duesler & Hendrickson, 1979; Richardson, Hatfield, Stoklosa & Wasson, 1973). In this paper we

describe the structure of *catena- μ -perchlorato- O,O' -tris- μ -quinoxaline- N,N' -dicopper(I) perchlorate*, where the quinoxaline ligands form bridges between the two copper atoms, and its IR spectrum.

Experimental. $\text{Cu}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ (0.922 g, 0.0025 mol) in ethanol (5 cm³) was added to quinoxaline (0.664 g, 0.0051 mol) also in ethanol (5 cm³). Bright red crystals separated after two hours reflux. These were filtered, washed with ethanol and ether and dried in air. The product is very stable and insoluble in all other solvents tried, except mineral acids.

The crystal selected for the intensity-data collection had dimensions 0.2 × 0.2 × 0.4 mm. Density by flotation. The unit-cell parameters were determined by least-squares treatment of the adjusted angular settings of 25 reflections measured on a Nicolet P3F diffractometer. The intensity measurements were carried out at room temperature (295 K) with graphite-monochromatized Mo $K\alpha$ radiation and the ω - 2θ scan technique. The scan rate varied from 2.5 to 29.3° min⁻¹, depending on the number of counts measured in a fast preliminary scan through the peak.

A set of 3074 unique reflections was obtained from 3266 reflections measured from $2\theta = 3$ –55° (h 0→12, k 0→19, l -13→13). 2520 reflections with $F_o > 6\sigma(F_o)$ were considered as observed and used in refinement. Three strong reflections (210, 303 and 412) monitored

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periodically during data collection exhibited no significant intensity variation. The intensities were corrected for Lorentz and polarization effects and also for absorption by the φ -scan technique. Max. and min. transmission factors 1.000, 0.851.

Systematic absences: hkl , $h0l$, no conditions; $0k0$, $k=2n+1$ and structure analysis established the space group as monoclinic $P2_1$ (No. 4). The structure was solved by direct and Fourier methods. Calculations were carried out on a Univac 1100/61 El computer using the programs *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) and *MPLN* (Truter & Vickerey, 1973). The neutral-atom scattering factors used were those of Cromer & Mann (1968). Real and imaginary anomalous-dispersion corrections were included for all non-H atoms (*International Tables for X-ray Crystallography*, 1974). At the end of the calculations an extinction correction was also applied but it proved to be unnecessary. All non-H atoms were located after successful Fourier syntheses.

The H atoms of the three quinoxaline molecules were located with fixed bond lengths (1.00 Å). The temperature factor of the H atoms was fixed as 0.05 Å². Anisotropic full-matrix least-squares refinement on F of non-H atoms yielded $R=0.048$ and $wR=0.049$, where $w=1/\sigma(F_o^2)$. After the last cycle the average shift/e.s.d. was 0.1576 and maximum shift/e.s.d. 1.790. The high value of $(\Delta/\sigma)_{\max}$ is due to that for Cu(1), U_{22} , $\Delta/\sigma=1.0221$; U_{13} , $\Delta/\sigma=1.5405$; Cu(2), U_{13} , $\Delta/\sigma=1.7898$. Δ/σ values of all other anisotropic temperature parameters of the heavy atoms were only some tenths or hundredths and changes in the coordinates in the fifth or sixth decimal place. A final difference map was practically featureless, with max. and min. heights +0.85 and -0.48 e Å⁻³. The origin of the space group ($P2_1$) was defined by the program *MULTAN80* using as origin-fixing reflections (hkl) 3,0,10, 295 and 103.

Infrared spectra were recorded in KBr disks on a Perkin-Elmer 577 Grating Infrared Spectrophotometer.

Discussion. The atomic coordinates and equivalent isotropic values of the anisotropic temperature-factor coefficients (Hamilton, 1959) for the non-H atoms are listed in Table 1.* The bond lengths and angles are given in Table 2.

The bond distances and angles of the quinoxaline molecules are comparable with earlier values measured in quinoxalinium perchlorate (Lipkowski, Andreotti &

Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\times 10^4$) of the non-H atoms with e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}(\text{Å}^2)$
Cu(1)	5136 (1)	7585 (1)	1458 (1)	392 (5)
Cu(2)	9096 (1)	6444 (1)	7370 (1)	329 (5)
Cl(1)	8350 (2)	8288 (2)	-465 (2)	368 (11)
Cl(2)	3871 (3)	5399 (2)	5162 (3)	506 (14)
O(1)	9881 (9)	8317 (7)	-94 (8)	882 (55)
O(2)	7474 (8)	8325 (6)	661 (6)	784 (52)
O(3)	7912 (8)	7524 (6)	-1255 (8)	841 (54)
O(4)	7954 (12)	9051 (7)	-1273 (13)	1336 (89)
O(5)	5332 (8)	5056 (6)	5303 (7)	734 (54)
O(6)	3497 (11)	5772 (7)	3899 (10)	1236 (80)
O(7)	3781 (18)	6067 (11)	6135 (15)	2329 (166)
O(8)	2907 (11)	4746 (8)	5378 (15)	1889 (112)
N(1)	6293 (7)	7190 (4)	3147 (6)	259 (33)
N(2)	8058 (7)	6794 (4)	5579 (6)	282 (35)
N(3)	4058 (7)	8833 (5)	1824 (6)	297 (36)
N(4)	2127 (7)	10346 (4)	1993 (6)	296 (35)
N(5)	3742 (7)	7052 (5)	-4 (7)	336 (37)
N(6)	1133 (7)	6625 (4)	-1684 (6)	281 (35)
C(1)	6092 (8)	7621 (6)	4295 (7)	345 (42)
C(2)	6981 (9)	7420 (6)	5526 (8)	372 (47)
C(3)	8277 (8)	6324 (5)	4401 (7)	259 (39)
C(4)	9451 (9)	5682 (5)	4393 (8)	337 (46)
C(5)	9702 (10)	5247 (6)	3201 (8)	383 (50)
C(6)	8778 (11)	5413 (7)	1993 (9)	451 (53)
C(7)	7634 (10)	6051 (6)	1977 (8)	370 (47)
C(8)	7390 (8)	6523 (6)	3195 (7)	274 (38)
C(9)	4427 (9)	9675 (6)	1474 (8)	369 (47)
C(10)	3494 (9)	10423 (6)	1585 (8)	315 (43)
C(11)	1685 (9)	9486 (5)	2380 (7)	261 (39)
C(12)	250 (9)	9349 (6)	2845 (8)	303 (42)
C(13)	-160 (9)	8498 (6)	3278 (8)	335 (45)
C(14)	835 (10)	7763 (6)	3269 (9)	449 (53)
C(15)	2268 (10)	7875 (5)	2813 (8)	353 (46)
C(16)	2689 (8)	8731 (5)	2322 (7)	274 (40)
C(17)	2866 (9)	6358 (6)	259 (8)	361 (44)
C(18)	1549 (9)	6144 (6)	-597 (8)	363 (47)
C(19)	2078 (8)	7311 (5)	-2033 (7)	269 (40)
C(20)	1739 (9)	7822 (5)	-3236 (7)	332 (44)
C(21)	2696 (10)	8508 (6)	-3559 (8)	382 (48)
C(22)	3996 (10)	8729 (6)	-2720 (9)	425 (51)
C(23)	4324 (9)	8267 (6)	-1551 (8)	351 (45)
C(24)	3385 (8)	7530 (6)	-1187 (7)	276 (38)

Sgarabotto, 1977). The free perchlorate ion has one short bond Cl(2)—O(8) [1.314 (12) Å] and the copper-coordinated one a longer bond Cl(1)—O(2) [1.416 (7) Å].

An *ORTEP* drawing (Johnson, 1965) in Fig. 1 shows the complex unit. As is normal with strong π acceptors, Cu^I atoms are four coordinated. The coordination polyhedron around both Cu atoms is a distorted tetrahedron. The Cu atoms have one long bond [2.385 (8) and 2.536 (8) Å] to an oxygen atom of the perchlorate ion and three shorter bonds [1.982 (6)—2.104 (7) Å] to the N atoms of the quinoxaline ligands (Table 2). The Cu—N and Cu—O bond distances in the coordination polyhedron are normal compared with similar copper complexes (Morosin *et al.*, 1975; Lumme *et al.*, 1987; Darriet *et al.*, 1979). The longer Cu—O bond distances are due to steric hindrances caused by the large quinoxaline molecules.

The title compound forms an infinite polymer, where all three quinoxaline molecules and one of the two perchlorate ions form bridges between Cu atoms (Fig. 2).

* Lists of structure factors, anisotropic thermal parameters, H-atom positions, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44133 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (Å) and angles (°)

The coordination sphere of copper atoms			
Cu(1)—N(1)	1.982 (6)	N(1)—Cu(1)—N(3)	108.7 (2)
Cu(1)—N(3)	2.104 (7)	N(1)—Cu(1)—N(5)	139.1 (3)
Cu(1)—N(5)	1.993 (7)	N(1)—Cu(1)—O(2)	89.9 (2)
Cu(1)—O(2)	2.536 (8)	N(3)—Cu(1)—N(5)	101.1 (3)
		N(3)—Cu(1)—O(2)	95.0 (3)
		N(5)—Cu(1)—O(2)	115.1 (2)
The quinoxaline molecules			
(1)			
N(1)—C(1)	1.325 (10)	C(1)—N(1)—C(8)	116.7 (6)
C(1)—C(2)	1.432 (10)	N(1)—C(1)—C(2)	122.0 (7)
C(2)—N(2)	1.325 (10)	C(1)—C(2)—N(2)	121.8 (7)
N(2)—C(3)	1.381 (9)	C(2)—N(2)—C(3)	117.4 (6)
C(3)—C(4)	1.408 (11)	N(2)—C(3)—C(4)	119.4 (6)
C(3)—C(8)	1.411 (10)	N(2)—C(3)—C(8)	120.4 (7)
C(4)—C(5)	1.375 (11)	C(8)—C(3)—C(4)	120.1 (7)
C(5)—C(6)	1.420 (11)	C(3)—C(4)—C(5)	119.0 (7)
C(6)—C(7)	1.382 (12)	C(4)—C(5)—C(6)	121.4 (7)
C(7)—C(8)	1.422 (11)	C(5)—C(6)—C(7)	120.3 (8)
C(8)—N(1)	1.380 (10)	C(6)—C(7)—C(8)	118.7 (7)
		C(7)—C(8)—N(1)	118.0 (6)
		C(7)—C(8)—C(3)	120.4 (7)
		C(3)—C(8)—N(1)	121.6 (7)
(2)			
N(3)—C(9)	1.324 (11)	C(9)—N(3)—C(16)	116.5 (7)
C(9)—C(10)	1.383 (12)	N(3)—C(9)—C(10)	123.0 (8)
C(10)—N(4)	1.324 (10)	C(9)—C(10)—N(4)	122.4 (8)
N(4)—C(11)	1.378 (10)	C(10)—N(4)—C(11)	117.1 (7)
C(11)—C(12)	1.413 (12)	N(4)—C(11)—C(12)	120.5 (7)
C(11)—C(16)	1.425 (11)	N(4)—C(11)—C(16)	119.7 (7)
C(12)—C(13)	1.372 (12)	C(16)—C(11)—C(12)	119.8 (7)
C(13)—C(14)	1.394 (12)	C(11)—C(12)—C(13)	120.2 (8)
C(14)—C(15)	1.404 (13)	C(12)—C(13)—C(14)	120.4 (8)
C(15)—C(16)	1.402 (11)	C(13)—C(14)—C(15)	120.9 (8)
C(16)—N(3)	1.366 (10)	C(14)—C(15)—C(16)	119.6 (7)
		C(15)—C(16)—N(3)	119.8 (7)
		C(15)—C(16)—C(11)	119.0 (7)
		C(11)—C(16)—N(3)	121.2 (7)
(3)			
N(5)—C(17)	1.319 (11)	C(17)—N(5)—C(24)	117.0 (7)
C(17)—C(18)	1.431 (11)	N(5)—C(17)—C(18)	121.7 (7)
C(18)—N(6)	1.314 (10)	C(17)—C(18)—N(6)	122.4 (8)
N(6)—C(19)	1.372 (10)	C(18)—N(6)—C(19)	117.1 (6)
C(19)—C(20)	1.418 (10)	N(6)—C(19)—C(20)	120.0 (6)
C(19)—C(24)	1.420 (10)	N(6)—C(19)—C(24)	120.7 (6)
C(20)—C(21)	1.371 (12)	C(24)—C(19)—C(20)	119.3 (7)
C(21)—C(22)	1.412 (12)	C(19)—C(20)—C(21)	118.9 (7)
C(22)—C(23)	1.352 (12)	C(20)—C(21)—C(22)	122.0 (7)
C(23)—C(24)	1.428 (12)	C(21)—C(22)—C(23)	120.2 (8)
C(24)—N(5)	1.379 (10)	C(22)—C(23)—C(24)	120.0 (7)
		C(23)—C(24)—N(5)	119.6 (6)
		C(23)—C(24)—C(19)	119.6 (7)
		C(19)—C(24)—N(5)	120.8 (7)
The perchlorate ions			
Cl(1)—O(1)	1.391 (8)	O(1)—Cl(1)—O(2)	112.8 (4)
Cl(1)—O(2)	1.416 (7)	O(1)—Cl(1)—O(3)	113.5 (5)
Cl(1)—O(3)	1.399 (9)	O(1)—Cl(1)—O(4)	109.0 (6)
Cl(1)—O(4)	1.399 (11)	O(2)—Cl(1)—O(3)	108.9 (5)
		O(2)—Cl(1)—O(4)	106.9 (6)
		O(3)—Cl(1)—O(4)	105.4 (6)
Cl(2)—O(5)	1.397 (8)	O(5)—Cl(2)—O(6)	112.9 (6)
Cl(2)—O(6)	1.381 (10)	O(5)—Cl(2)—O(7)	106.9 (7)
Cl(2)—O(7)	1.377 (16)	O(5)—Cl(2)—O(8)	110.3 (6)
Cl(2)—O(8)	1.314 (12)	O(6)—Cl(2)—O(7)	109.5 (8)
		O(6)—Cl(2)—O(8)	108.6 (7)
		O(7)—Cl(2)—O(8)	108.8 (9)

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

No hydrogen bonds of the type C—H...O exist, if for the C(—H)...O distance the contact criterion ≤ 3.2 Å is accepted (Taylor & Kennard, 1982).

Least-squares calculations of the planes through the three quinoxaline ligands showed them to be practically planar; maximum atom deviations are 0.043 (9) of C(5), -0.059 (6) of N(3) and 0.038 (3) Å of N(6) for quinoxaline (1), (2) and (3), respectively. One of the quinoxaline molecules is nearly perpendicular to two other molecules; the angles between the molecular planes are 86.9 (2) and 96.9 (1)° between (1) and (2), and between (1) and (3), respectively. The angle between the quinoxaline molecules (2) and (3) is only 61.1 (2)°.

The infrared spectrum ($4000\text{--}250\text{ cm}^{-1}$) is very similar to those obtained earlier for cobalt(II) complexes with quinoxaline (Billing, 1969) and shows in most cases the same bands as quinoxaline, but weakened and shifted to some extent, pointing to coordination to the Cu atoms.

The most important bands shown by the present complex are the following: 3050w, 1495s; 1460, 1455w; 1415m; 1365, 1355m; 1280w; 1210, 1200w;

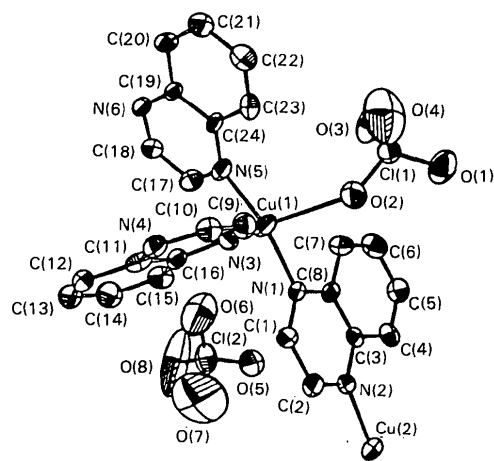


Fig. 1. ORTEP (Johnson, 1965) drawing with the numbering scheme of the $[\text{Cu}_2(\text{C}_8\text{H}_6\text{N}_2)_3(\text{ClO}_4)_2]$ complex. (50% probability ellipsoids are shown for the non-H atoms.)

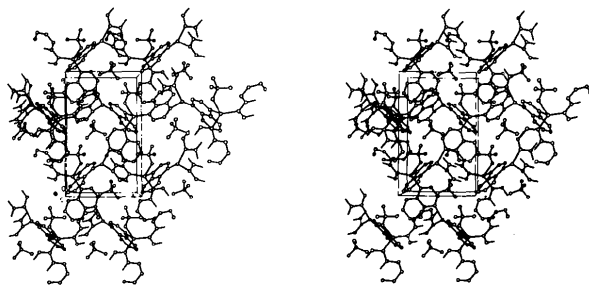


Fig. 2. Stereoview of the packing of the molecules as viewed down *c*.

1140, 1110, 1090 ν s; 1035, 1020 m ; 960, 955, 950 m ; 885 m , 860 s , 755 ν s; 635, 625 m ; 410, 400 m ; 275 w . Among these the band at 860 cm^{-1} seems to be specially characteristic of the complex; this is absent from the spectrum of quinoxaline. The 275 cm^{-1} band we assign to $\nu(\text{Cu}-\text{N})$ stretching vibrations.

The IR spectrum of the complex shows the strong band found at 953 cm^{-1} in the quinoxaline spectrum (Billing, 1969), obviously in the region 1000–900 cm^{-1} , split into a triplet at 960–950 cm^{-1} and pointing to quinoxaline coordination. A complex studied earlier, triaquabis(quinoxaline)copper(II) perchlorate, with terminal quinoxaline ligands, has a medium peak at 940 cm^{-1} in its IR spectrum (Lumme *et al.*, 1987). The responsibility of this band for the coordination of pyrazine to the central atom has earlier been assumed (Lever, Lewis & Nyholm, 1962). The present complex also shows three intense bands of the bridging bidentate perchlorate in the range 1150–1050 cm^{-1} (1140, 1110 and 1090 cm^{-1}) owing to $\nu(\text{Cl}-\text{O})$ stretching vibrations.

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Structure of {4,4'-[Iminobis(1,3-propanediylnitrilo)]-di-2-pentanonato-*N,N',N'',O,O'*} copper

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Abstract. $[\text{Cu}(\text{C}_{16}\text{H}_{27}\text{N}_3\text{O}_2)]$, $M_r = 356.95$, monoclinic, $C2/c$, $a = 22.234$ (5), $b = 11.753$ (5), $c = 19.005$ (4) Å, $\beta = 135.57$ (2)°, $V = 3476.8$ Å³, $Z = 8$, $D_x = 1.364$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 12.7$ cm⁻¹, $F(000) = 1512$, $T = 168$ K, final $R = 0.036$ for 2813 unique observed reflections. The copper coordination is fivefold, approximately trigonal bipyramidal. Imine nitrogens occupy *trans* apical positions (1.96 Å, 175°). Two oxygens (1.98 Å, 142°) and the secondary nitrogen of the pentadentate ligand (2.29 Å, 103°) occupy the trigonal equatorial sites.

Introduction. Since initial isolation, neutral five-coordinate Cu^{II} complexes containing doubly condensed pentadentate Schiff bases have been described as most probably possessing distorted square-pyramidal

structure (Cummings & Wallis, 1974; Cummings, Chen, Chu, McKinney & Willis, 1981; Cummings, Braydich & Fortman, 1983). The structure of the title compound demonstrates that in the solid state the molecule is more appropriately described as trigonal bipyramidal with a nearly perfect twofold axis along the copper–secondary-amine vector.

Experimental. Green crystals were obtained following method *B* of Cummings *et al.* (1981). Nearly cube-shaped specimen, dimensions 0.30 × 0.30 × 0.40 mm. Enraf–Nonius CAD-4 diffractometer with graphite crystal monochromator using $\omega-2\theta$, $2\theta < 55^\circ$, scan speed varying from 2 to 20° min⁻¹ (in 2θ). Lattice parameters from 25 reflections in θ range of 7–11°. Empirical absorption corrections (Walker & Stuart,