# Crystal structure of diaquabis(ethylenediamine-$\kappa^2N,N'$)copper(II) bis(*p*-chlorobenzoate), $[Cu(C_2H_8N_2)_2(H_2O)_2](C_7H_4ClO_2)_2$

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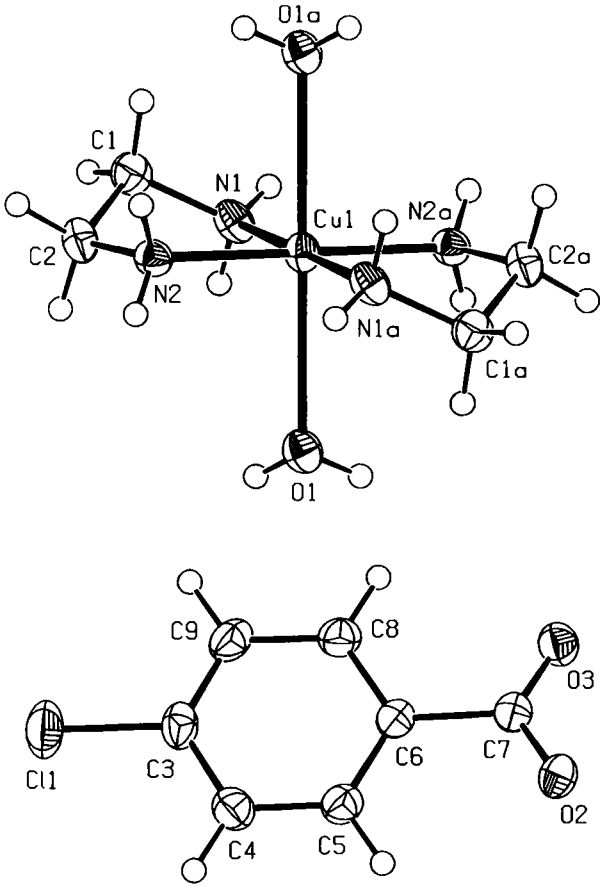


**Abstract**

$C_{18}H_{28}Cl_2CuN_4O_6$, orthorhombic, *Pbca* (no. 61), $a = 10.623(3)$ Å, $b = 7.187(3)$ Å, $c = 29.912(8)$ Å, $V = 2283.7$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.024$, $wR_{ref}(F) = 0.028$, $T = 173$ K.

**Source of material**

*p*-Chlorobenzoic acid was used as purchased (Wako Pure Chem.). All other chemicals were of analytical grade and were used without further purification. In a 50 ml beaker with vigor stirring, 2 M NaOH aqueous solution was added dropwise into *p*-chlorobenzoic acid (0.313 g, 2 mmol) suspended in 20 ml water until all the *p*-chlorobenzoic acid dissolved and the pH value kept at about 8. Then $CuCl_2 \cdot 2H_20$ (0.174 g, 1 mmol) in 5ml water was added into above solution slowly. The suspended liquid was stirred for 1 h at room temperature, and the precipitate was separated centrifugally and washed with water adequately. To the precipitate, ethylenediamine (0.180 g, 3 mmol) in 5 ml methanol solution was added, and a deep blue solution was obtained, which was allowed to keep in air and room temperature. Some blue well-shaped block-like single crystals appeared after two days.

Elemental analysis: found – C, 40.29 %; H, 5.81 %; N, 10.5 8 %; calc. for $C_{18}H_{28}Cl_2CuN_4O_6$ – C, 40.72 %; H, 5.32 %; N, 10.55 %.

**Discussion**

It is well-known that copper(II) complexes having ethylenediamine ligands show flexibility of their coordination environment and adopt semi-coordination with tetragonal distortion. Hitherto, there are several complex compounds from benzoate and its derivates being synthesized, such as diaquabis(ethylenediamine-$\kappa^2N,N'$)copper(II) bis(4-fluorobenzoate) [1], *trans*-bis(ethylenediamine)bis(*p*-nitrobenzoxasulfamato)-copper(II) [2] and bis-(ethylenediamine)copper(II) benzoylacetonate dihydrate [3]. In continuation of our studies on salts of copper complex cations with organic acids, the crystal structure of 4-chlorobenzoate of a Cu(II) complex incorporating aqua and ethylenediamine ligands is presented here.

The complex consists of Cu(II) in a distorted octahedral coordination environment composed of two oxygen donors from two water molecules and four amine donors from two ethylenediamine molecules. In the $CuN_4O_2$ octahedron, the Cu—N distances range from 2.009(2) Å to 2.016(2) Å, with a small N–Cu–N angle of 84.30(6)°, being in the normal range, but a Cu—O distance of 2.618(1) Å shows that the water molecule is weakly coordinated to Cu(II) in an axial position, which is consistent to the representation of diaquabis(ethylenediamine-$\kappa^2N,N'$)copper(II) bis(4-fluorobenzoate) [1]. All values of the anionic part of the title structure are normal [4-10]. The molecular packing shows that conventional intramolecular and intermolecular O–H···O and N–H···O hydrogen bonds occur as expected, linking the cations and anions into a three-dimensional network.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | blue block, size 0.20 × 0.30 × 0.60 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 12.32 cm$^{-1}$ |
| Diffractometer, scan mode: | Rigaku R-AXIS RAPID, $\omega/\varphi$ |
| $2\theta_{max}$: | 54.96° |
| $N(hkl)_{measured}$, $N(hki)_{unique}$: | 22421, 2616 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 3\,\sigma(I_{obs})$, 1552 |
| $N(param)_{refined}$: | 142 |
| Programs: | SIR92 [11], CRYSTALS [12], PLATON [13] |

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**Table 2.** Atomic coordinates and displacement parameters (in $Å^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1) | 8c | –0.1085 | 0.2760 | 0.5521 | 0.050 |
| H(2) | 8c | 0.1084 | 0.7021 | 0.5566 | 0.050 |
| H(3) | 8c | 0.1768 | 0.5326 | 0.5557 | 0.050 |
| H(5) | 8c | –0.2023 | 0.3977 | 0.5369 | 0.050 |
| H(6) | 8c | 0.0886 | 0.1041 | 0.4735 | 0.050 |
| H(7) | 8c | 0.1111 | 0.1054 | 0.5177 | 0.050 |
| H(8) | 8c | –0.1529 | 0.4474 | 0.6115 | 0.036 |
| H(9) | 8c | –0.1402 | 0.6383 | 0.5823 | 0.036 |
| H(10) | 8c | 0.0509 | 0.6033 | 0.6204 | 0.035 |
| H(11) | 8c | 0.0621 | 0.3868 | 0.6043 | 0.035 |
| H(12) | 8c | 0.2746 | –0.0344 | 0.7259 | 0.038 |
| H(13) | 8c | 0.2382 | –0.0837 | 0.6474 | 0.035 |
| H(14) | 8c | –0.1158 | 0.1470 | 0.6595 | 0.037 |
| H(15) | 8c | –0.0776 | 0.2049 | 0.7375 | 0.040 |

**Table 3.** Atomic coordinates and displacement parameters (in $Å^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| Cu(1) | 4a | 0 | ½ | ½ | 0.0177(1) | 0.0273(1) | 0.0221(1) | –0.0040(2) | –0.0009(1) | 0.0003(1) |
| O(1) | 8c | 0.1128(1) | 0.1767(2) | 0.49530(4) | 0.0291(6) | 0.0324(7) | 0.0265(6) | –0.0016(5) | –0.0008(6) | 0.0044(5) |
| N(1) | 8c | –0.1253(1) | 0.4023(2) | 0.54451(5) | 0.0181(7) | 0.0328(9) | 0.0298(7) | –0.0040(7) | –0.0035(6) | 0.0051(6) |
| N(2) | 8c | 0.0995(1) | 0.5727(2) | 0.55465(5) | 0.0198(7) | 0.0246(7) | 0.0271(7) | –0.0006(6) | –0.0026(6) | –0.0007(6) |
| C(1) | 8c | –0.1074(2) | 0.5089(3) | 0.58625(6) | 0.0293(9) | 0.034(1) | 0.0262(8) | 0.001(1) | 0.0040(7) | 0.0041(8) |
| C(2) | 8c | 0.0329(2) | 0.5140(3) | 0.59553(6) | 0.0327(9) | 0.0285(9) | 0.0250(8) | –0.0041(9) | –0.0051(6) | 0.0038(8) |
| Cl(1) | 8c | 0.12830(6) | 0.13196(9) | 0.79330(2) | 0.0628(3) | 0.0534(3) | 0.0243(2) | –0.0008(3) | –0.0022(2) | –0.0059(2) |
| O(2) | 8c | 0.1318(1) | –0.0329(2) | 0.57338(4) | 0.0305(6) | 0.0310(7) | 0.0247(6) | –0.0028(6) | 0.0048(5) | –0.0012(5) |
| O(3) | 8c | –0.0727(1) | 0.0231(2) | 0.58291(4) | 0.0305(7) | 0.0371(8) | 0.0270(6) | 0.0011(6) | –0.0004(5) | 0.0013(6) |
| C(3) | 8c | 0.1015(2) | 0.0910(3) | 0.73649(6) | 0.045(1) | 0.026(1) | 0.0206(8) | –0.0046(9) | 0.0007(8) | 0.0014(7) |
| C(4) | 8c | 0.1941(2) | 0.0059(3) | 0.71157(6) | 0.0341(9) | 0.032(1) | 0.0299(8) | 0.001(1) | –0.0031(7) | 0.0028(9) |
| C(5) | 8c | 0.1724(2) | –0.0230(3) | 0.66629(6) | 0.0315(9) | 0.027(1) | 0.0281(8) | 0.0015(8) | 0.0036(7) | 0.0003(8) |
| C(6) | 8c | 0.0596(2) | 0.0311(2) | 0.64683(6) | 0.0298(9) | 0.020(1) | 0.0231(7) | –0.0020(7) | 0.0042(7) | 0.0021(7) |
| C(7) | 8c | 0.0372(2) | 0.0044(3) | 0.59730(6) | 0.0302(8) | 0.0194(7) | 0.0238(7) | –0.0036(9) | 0.0017(6) | 0.0018(8) |
| C(8) | 8c | –0.0333(2) | 0.1124(3) | 0.67325(6) | 0.032(1) | 0.028(1) | 0.0294(9) | 0.0038(8) | 0.0032(7) | 0.0024(8) |
| C(9) | 8c | –0.0122(2) | 0.1447(3) | 0.71832(6) | 0.041(1) | 0.0292(9) | 0.0278(9) | 0.0035(9) | 0.0099(8) | –0.0012(7) |

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