Z. Kristallogr. NCS 221 (2006) 489-491 / DOI 10.1524/ncrs.2006.0168 



# Crystal structure of 2-($\mu$-2-oxyphenyl)-1-(2-($\mu$-2-oxybenzyl)aminoethyl)-3-(2-(2-oxybenzyl)-bis(aminoethyl))imidazolidine-dicopper(II) nitrate trihydrate, $[Cu_2(C_{29}H_{32}N_5O_3)][NO_3] \cdot 3H_2O$

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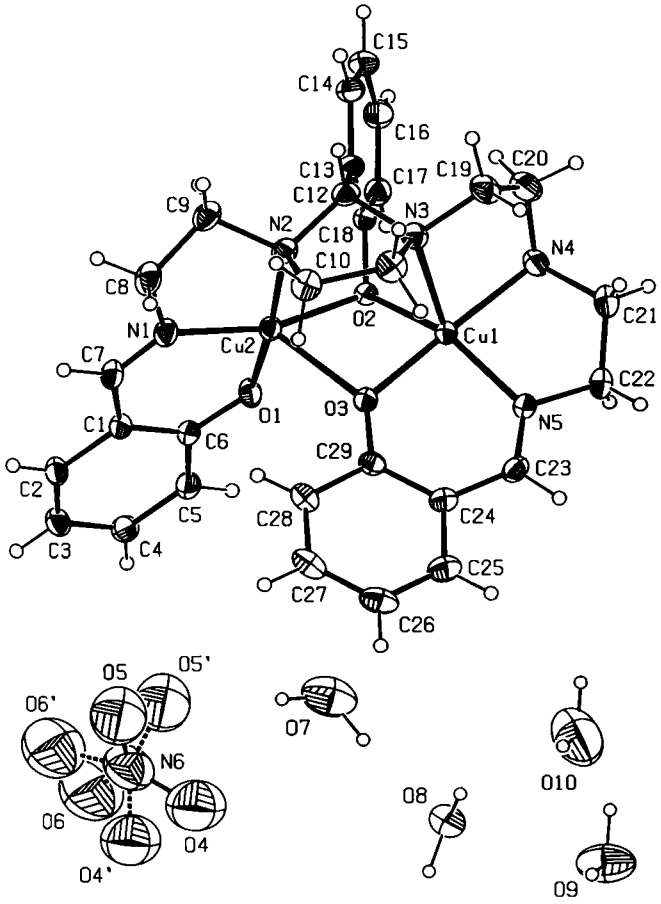
Received August 23, 2006, accepted and available on-line November 16, 2006; CCDC no. 1267/1879




**Abstract**

$C_{29}H_{38}Cu_2N_6O_9$, monoclinic, $C12/c1$ (no. 15), $a$ = 21.999(1) Å, $b$ = 18.255(1) Å, $c$ = 18.333(1) Å, $\beta$ = 121.959(1)°, $V$ = 6246.0 Å$^3$, $Z$ = 8, $R_{gt}(F)$ = 0.035, $wR_{ref}(F^2)$ = 0.105, $T$ = 295 K.

**Source of material**

The title compound was obtained by adding 10 mL of an aqueous solution containing 0.5 mmol $Cu(NO_3)_2$ to an ethanol solution (15 mL) of 1 mmol tetraethylenepentamine and 2 mmol salicylaldehyde, then kept in reflux at 333 K for 3 h. The dark blue solution was allowed to concentrate slowly at room temperature, the resulting blue block-like crystals suitable for X-ray analysis were isolated after two weeks.

Elemental analysis – found: C, 46.93 %; H, 5.20 %; N, %11.32 %; calc. for $C_{29}H_{38}N_6Cu_2O_9$: C, 46.96 %; H, 5.16 %; N, 11.33 %.

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**Discussion**

Dinuclear copper(II) unit exists on active sites of many metalloenzymes and metal-proteins, for example, hemocyanin, tyrosinase, cytochrome oxidase etc. [1]. Due to the magnetic interaction from their electrotransfer it has an important effect on the physiological and catalyzing activity of organisms. There has been increasing interest in the design and syntheses of dinuclear copper(II) complexes, and several dinuclear copper(II) complexes have been reported [2-9], Schiff base complexes containing salicylaldehyde and amine derivatives have been also often reported [10-13]. The complexes of salicylaldehyde with polyamines and bis(phenoxo) bridged dinuclear copper(II) complexes are sparse [14].

X-ray crystal structure analysis has shown that the title complex is composed of dinuclear copper(II) unit, nitrate anion and crystal water. In the dinuclear copper(II) unit the tetraethylenepentamine is joining salicylaldehyde molecules forming a bis-Schiff base. The bond distances N1—C7 and N5—C23 are 1.286(3) Å and 1.281(3) Å, respectively, which are obviously double bonds. The N3—C12 distance of 1.482(3) Å represents a single bond. Two copper(II) ions are held together by a phenolic hydroxyl bridge. The Cu···Cu distance is 3.088 Å. The environments of the two Cu(II) centers are different. The Cu2 atom is five-coordinated in a distorted trigonal bipyramidal environment. It is coordinated by three oxygen (O1, O2, O3) and two nitrogen atoms (N1, N2). The axis of the polyhedron is formed by O1–Cu2–N2. The bond distances Cu2—O1 and Cu2—N2 are 1.917(2) Å and 2.112(2) Å, respectively. The equatorial plane is made up of O2, O3, N1. The corresponding bond distances Cu2—O2, Cu2—O3 and Cu2—N1 are 1.982(2) Å, 2.299(2) Å and 1.929(2) Å, respectively. The Cu1 atom shows a five-coordinated distorted square-pyramidal environment formed by two O (O2, O3) and three N atoms (N3, N4, N5). The apical Cu1—O2 bond is 2.018(2) Å. The basal plane is made up of O3, N3, N4 and N5. The bond lengths Cu1—O3, Cu1—N3, Cu1—N4 and Cu1—N5 are 1.927(2) Å, 2.271(2) Å, 2.047(2) Å and 1.938(2) Å, respectively. There are several hydrogen bonds in the structure: (1) hydrogen bonds of the O atom from nitrate with crystal water; (2) O atom from phenolic hydroxyl with crystal water; (3) H atoms from the crystal water with O atoms from the crystal water. The range of the bond distances for O10–H6W···O5′, O8–H4W···O1, O8–H3W···O7″, O7–H2W···O4‴ and O7–H1W···O5 is 1.94 Å – 2.70 Å. In addition, there are weak $\pi$-$\pi$ interactions between phenyl rings of neighbored molecules, the distances of phenyl rings C1A – C6A with rings C1B – C6B is 3.540 Å, it is close to 3.532 Å which was reported in [14], the dihedral angle of phenyl rings is 0°. The crystal structure is stabilized by these weak interactions to form three-dimensional network, the free water molecules and nitrate ions are resided into the cavities of the framework.

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

| | |
|---|---|
| Crystal: | blue block, size 0.35 × 0.41 × 0.50 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 14.25 $cm^{-1}$ |
| Diffractometer, scan mode: | Bruker SMART CCD, $\varphi/\omega$ |
| $2\theta_{max}$: | 54.98° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 26741, 7160 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 6235 |
| $N(param)_{refined}$: | 426 |
| Programs: | SHELXS-97 [15], SHELXL-97 [16], SHELXTL [17] |

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

| Atom | Site | x | y | z | $U_{iso}$ |
|---|---|---|---|---|---|
| H(4N) | 8*f* | 0.1300 | 0.3280 | 0.3317 | 0.049 |
| H(2) | 8*f* | 0.6454 | 0.4264 | 0.5821 | 0.050 |
| H(3) | 8*f* | 0.6828 | 0.4543 | 0.7215 | 0.054 |
| H(4) | 8*f* | 0.5994 | 0.4568 | 0.7632 | 0.052 |
| H(5) | 8*f* | 0.4812 | 0.4315 | 0.6664 | 0.048 |
| H(7) | 8*f* | 0.5574 | 0.3991 | 0.4396 | 0.047 |
| H(8A) | 8*f* | 0.4631 | 0.3127 | 0.2992 | 0.059 |
| H(8B) | 8*f* | 0.4755 | 0.3969 | 0.2934 | 0.059 |
| H(9A) | 8*f* | 0.3554 | 0.4224 | 0.2349 | 0.053 |
| H(9B) | 8*f* | 0.3575 | 0.3557 | 0.1819 | 0.053 |
| H(10A) | 8*f* | 0.3702 | 0.2227 | 0.3068 | 0.053 |
| H(10B) | 8*f* | 0.3486 | 0.2386 | 0.2114 | 0.053 |
| H(11A) | 8*f* | 0.2374 | 0.1999 | 0.1601 | 0.055 |
| H(11B) | 8*f* | 0.2635 | 0.1711 | 0.2534 | 0.055 |
| H(12) | 8*f* | 0.2341 | 0.3403 | 0.1518 | 0.043 |
| H(14) | 8*f* | 0.1756 | 0.4526 | 0.1133 | 0.051 |
| H(15) | 8*f* | 0.1266 | 0.5536 | 0.1394 | 0.060 |
| H(16) | 8*f* | 0.1476 | 0.5729 | 0.2752 | 0.057 |
| H(17) | 8*f* | 0.2199 | 0.4932 | 0.3866 | 0.046 |
| H(19A) | 8*f* | 0.1295 | 0.2154 | 0.1713 | 0.055 |
| H(19B) | 8*f* | 0.1260 | 0.2859 | 0.1202 | 0.055 |
| H(20A) | 8*f* | 0.1096 | 0.3604 | 0.2101 | 0.067 |
| H(20B) | 8*f* | 0.0546 | 0.2958 | 0.1836 | 0.067 |
| H(21A) | 8*f* | 0.0632 | 0.2398 | 0.3219 | 0.075 |
| H(21B) | 8*f* | 0.0898 | 0.1903 | 0.2745 | 0.075 |
| H(22A) | 8*f* | 0.1377 | 0.1370 | 0.3992 | 0.058 |
| H(22B) | 8*f* | 0.1543 | 0.2116 | 0.4491 | 0.058 |
| H(23) | 8*f* | 0.2530 | 0.0967 | 0.4783 | 0.046 |
| H(25) | 8*f* | 0.3608 | 0.0301 | 0.5402 | 0.058 |
| H(26) | 8*f* | 0.4765 | 0.0132 | 0.5747 | 0.066 |
| H(27) | 8*f* | 0.5308 | 0.1029 | 0.5388 | 0.060 |
| H(28) | 8*f* | 0.4725 | 0.2099 | 0.4740 | 0.050 |
| H(1W) | 8*f* | 0.7440 | 0.3822 | 0.4945 | 0.054 |
| H(2W) | 8*f* | 0.6788 | 0.3353 | 0.4507 | 0.058 |
| H(3W) | 8*f* | 0.3274 | 0.5436 | 0.5490 | 0.087 |
| H(4W) | 8*f* | 0.3599 | 0.4773 | 0.5373 | 0.073 |
| H(5W) | 8*f* | 0.0000 | 0.4246 | 0.2090 | 0.059 |
| H(6W) | 8*f* | 0.0001 | 0.5853 | 0.2097 | 0.054 |

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

| Atom | Site | Occ. | x | y | z | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| N(6) | 8*f* | 0.630(7) | 0.8890(4) | 0.3103(4) | 0.5683(4) | 0.153(5) | 0.146(5) | 0.174(5) | 0.045(4) | 0.087(4) | 0.002(4) |
| O(4) | 8*f* | 0.630 | 0.8300(6) | 0.2819(6) | 0.5130(7) | 0.221(7) | 0.196(6) | 0.202(6) | 0.020(6) | 0.104(5) | –0.021(5) |
| O(5) | 8*f* | 0.630 | 0.8872(6) | 0.3767(5) | 0.5657(8) | 0.194(7) | 0.162(6) | 0.241(7) | 0.016(5) | 0.104(5) | 0.017(5) |
| O(6) | 8*f* | 0.630 | 0.9326(7) | 0.2653(7) | 0.6111(9) | 0.240(7) | 0.226(8) | 0.279(7) | 0.064(6) | 0.119(5) | 0.008(6) |
| N(6′) | 8*f* | 0.370 | 0.8890(4) | 0.3103(4) | 0.5683(4) | 0.153(5) | 0.146(5) | 0.174(5) | 0.045(4) | 0.087(4) | 0.002(4) |
| O(4′) | 8*f* | 0.370 | 0.887(1) | 0.2620(9) | 0.523(1) | 0.221(7) | 0.196(6) | 0.202(6) | 0.020(6) | 0.104(5) | –0.021(5) |
| O(5′) | 8*f* | 0.370 | 0.8590(9) | 0.3544(9) | 0.588(1) | 0.194(7) | 0.162(6) | 0.241(7) | 0.016(5) | 0.104(5) | 0.017(5) |
| O(6′) | 8*f* | 0.370 | 0.9574(6) | 0.321(1) | 0.624(1) | 0.240(7) | 0.226(8) | 0.279(7) | 0.064(6) | 0.119(5) | 0.008(6) |
| Cu(1) | 8*f* | | 0.24489(1) | 0.27129(2) | 0.36797(2) | 0.0256(1) | 0.0317(2) | 0.0353(2) | –0.0020(1) | 0.0136(1) | 0.0059(1) |
| Cu(2) | 8*f* | | 0.37515(1) | 0.35947(2) | 0.39527(2) | 0.0288(1) | 0.0375(2) | 0.0284(1) | –0.0048(1) | 0.0168(1) | –0.0011(1) |
| O(1) | 8*f* | | 0.42018(8) | 0.3979(1) | 0.5097(1) | 0.0296(8) | 0.058(1) | 0.0328(8) | –0.0112(7) | 0.0179(7) | –0.0065(7) |
| O(2) | 8*f* | | 0.28038(8) | 0.37527(8) | 0.38131(9) | 0.0278(7) | 0.0307(7) | 0.0278(7) | –0.0014(6) | 0.0131(6) | 0.0015(6) |
| O(3) | 8*f* | | 0.34495(8) | 0.24682(9) | 0.4230(1) | 0.0296(8) | 0.0337(8) | 0.0421(9) | 0.0004(6) | 0.0179(7) | 0.0075(7) |
| O(7) | 8*f* | | 0.7106(2) | 0.3605(2) | 0.4994(3) | 0.175(4) | 0.111(3) | 0.108(3) | 0.056(3) | 0.077(3) | 0.026(2) |
| O(8) | 8*f* | | 0.3477(1) | 0.5006(1) | 0.5704(2) | 0.086(2) | 0.060(1) | 0.075(2) | 0.010(1) | 0.053(1) | 0.013(1) |
| O(9) | 4*e* | | 0 | 0.3981(3) | ¼ | 0.231(7) | 0.112(4) | 0.110(4) | 0 | 0.114(5) | 0 |
| O(10) | 4*e* | | 0 | 0.5566(4) | ¼ | 0.206(8) | 0.134(6) | 0.37(1) | 0 | 0.198(9) | 0 |
| N(1) | 8*f* | | 0.4596(1) | 0.3726(1) | 0.3897(1) | 0.037(1) | 0.047(1) | 0.038(1) | –0.0044(8) | 0.0261(9) | –0.0011(8) |
| N(2) | 8*f* | | 0.3282(1) | 0.3260(1) | 0.2661(1) | 0.040(1) | 0.035(1) | 0.0320(9) | –0.0035(8) | 0.0212(8) | –0.0009(8) |
| N(3) | 8*f* | | 0.2214(1) | 0.2740(1) | 0.2316(1) | 0.036(1) | 0.033(1) | 0.034(1) | –0.0047(8) | 0.0143(8) | –0.0039(8) |
| N(4) | 8*f* | | 0.1368(1) | 0.2891(1) | 0.3058(1) | 0.0300(9) | 0.042(1) | 0.042(1) | –0.0020(8) | 0.0135(9) | 0.0020(9) |
| N(5) | 8*f* | | 0.2266(1) | 0.1865(1) | 0.4172(1) | 0.0315(9) | 0.037(1) | 0.036(1) | –0.0050(8) | 0.0187(8) | 0.0021(8) |
| C(1) | 8*f* | | 0.5400(1) | 0.4093(1) | 0.5375(2) | 0.030(1) | 0.033(1) | 0.041(1) | –0.0034(8) | 0.0179(9) | 0.0047(9) |
| C(2) | 8*f* | | 0.6123(1) | 0.4266(1) | 0.5989(2) | 0.030(1) | 0.040(1) | 0.052(1) | –0.0037(9) | 0.020(1) | 0.004(1) |
| C(3) | 8*f* | | 0.6349(1) | 0.4438(1) | 0.6820(2) | 0.031(1) | 0.042(1) | 0.048(1) | –0.007(1) | 0.012(1) | 0.002(1) |
| C(4) | 8*f* | | 0.5846(1) | 0.4452(1) | 0.7068(2) | 0.043(1) | 0.043(1) | 0.035(1) | –0.011(1) | 0.014(1) | 0.001(1) |
| C(5) | 8*f* | | 0.5137(1) | 0.4297(1) | 0.6485(2) | 0.037(1) | 0.044(1) | 0.037(1) | –0.008(1) | 0.019(1) | 0.000(1) |
| C(6) | 8*f* | | 0.4886(1) | 0.4110(1) | 0.5622(2) | 0.031(1) | 0.033(1) | 0.034(1) | –0.0048(8) | 0.0164(9) | 0.0023(9) |
| C(7) | 8*f* | | 0.5216(1) | 0.3936(1) | 0.4516(2) | 0.035(1) | 0.045(1) | 0.047(1) | –0.004(1) | 0.027(1) | 0.002(1) |
| C(8) | 8*f* | | 0.4479(2) | 0.3615(2) | 0.3037(2) | 0.052(2) | 0.066(2) | 0.046(1) | –0.012(1) | 0.036(1) | –0.008(1) |
| C(9) | 8*f* | | 0.3686(2) | 0.3713(2) | 0.2382(2) | 0.056(2) | 0.051(2) | 0.037(1) | –0.010(1) | 0.031(1) | –0.002(1) |
| C(10) | 8*f* | | 0.3348(2) | 0.2457(2) | 0.2531(2) | 0.052(1) | 0.041(1) | 0.046(1) | –0.001(1) | 0.030(1) | –0.008(1) |
| C(11) | 8*f* | | 0.2606(2) | 0.2137(2) | 0.2201(2) | 0.052(2) | 0.037(1) | 0.048(1) | –0.004(1) | 0.026(1) | –0.010(1) |
| C(12) | 8*f* | | 0.2496(1) | 0.3404(1) | 0.2127(1) | 0.041(1) | 0.037(1) | 0.026(1) | –0.005(1) | 0.0148(9) | –0.0007(9) |
| C(13) | 8*f* | | 0.2260(1) | 0.4102(1) | 0.2333(2) | 0.035(1) | 0.031(1) | 0.032(1) | –0.0034(9) | 0.0132(9) | 0.0029(9) |
| C(14) | 8*f* | | 0.1841(1) | 0.4602(1) | 0.1681(2) | 0.042(1) | 0.042(1) | 0.035(1) | –0.005(1) | 0.013(1) | 0.007(1) |

**Table 3.** Continued.

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| C(15) | 8*f* | 0.1548(2) | 0.5209(2) | 0.1836(2) | 0.043(1) | 0.042(1) | 0.052(2) | 0.005(1) | 0.016(1) | 0.018(1) |
| C(16) | 8*f* | 0.1677(2) | 0.5325(2) | 0.2649(2) | 0.044(1) | 0.036(1) | 0.063(2) | 0.008(1) | 0.028(1) | 0.009(1) |
| C(17) | 8*f* | 0.2107(1) | 0.4843(1) | 0.3317(2) | 0.038(1) | 0.035(1) | 0.045(1) | 0.0002(9) | 0.023(1) | 0.002(1) |
| C(18) | 8*f* | 0.2398(1) | 0.4229(1) | 0.3165(1) | 0.027(1) | 0.028(1) | 0.034(1) | -0.0037(8) | 0.0132(8) | 0.0029(8) |
| C(19) | 8*f* | 0.1424(1) | 0.2668(2) | 0.1771(2) | 0.040(1) | 0.047(1) | 0.037(1) | -0.008(1) | 0.010(1) | -0.009(1) |
| C(20) | 8*f* | 0.1052(2) | 0.3081(2) | 0.2154(2) | 0.040(1) | 0.057(2) | 0.054(2) | 0.000(1) | 0.013(1) | 0.006(1) |
| C(21) | 8*f* | 0.1053(2) | 0.2245(2) | 0.3216(2) | 0.036(1) | 0.070(2) | 0.076(2) | -0.006(1) | 0.026(1) | 0.019(2) |
| C(22) | 8*f* | 0.1539(1) | 0.1869(2) | 0.4020(2) | 0.037(1) | 0.057(2) | 0.058(2) | -0.005(1) | 0.029(1) | 0.009(1) |
| C(23) | 8*f* | 0.2702(1) | 0.1346(1) | 0.4602(2) | 0.045(1) | 0.033(1) | 0.040(1) | -0.0051(9) | 0.024(1) | 0.0044(9) |
| C(24) | 8*f* | 0.3434(1) | 0.1297(1) | 0.4829(2) | 0.041(1) | 0.033(1) | 0.033(1) | 0.0031(9) | 0.018(1) | 0.0024(9) |
| C(25) | 8*f* | 0.3826(2) | 0.0661(2) | 0.5258(2) | 0.059(2) | 0.037(1) | 0.046(1) | 0.007(1) | 0.025(1) | 0.009(1) |
| C(26) | 8*f* | 0.4518(2) | 0.0557(2) | 0.5468(2) | 0.055(2) | 0.046(2) | 0.046(2) | 0.018(1) | 0.014(1) | 0.006(1) |
| C(27) | 8*f* | 0.4842(1) | 0.1099(2) | 0.5257(2) | 0.034(1) | 0.055(2) | 0.044(1) | 0.009(1) | 0.009(1) | -0.008(1) |
| C(28) | 8*f* | 0.4489(1) | 0.1740(2) | 0.4857(2) | 0.031(1) | 0.045(1) | 0.041(1) | 0.001(1) | 0.014(1) | -0.004(1) |
| C(29) | 8*f* | 0.3774(1) | 0.1854(1) | 0.4624(1) | 0.032(1) | 0.034(1) | 0.027(1) | 0.0010(8) | 0.0121(8) | -0.0019(8) |

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