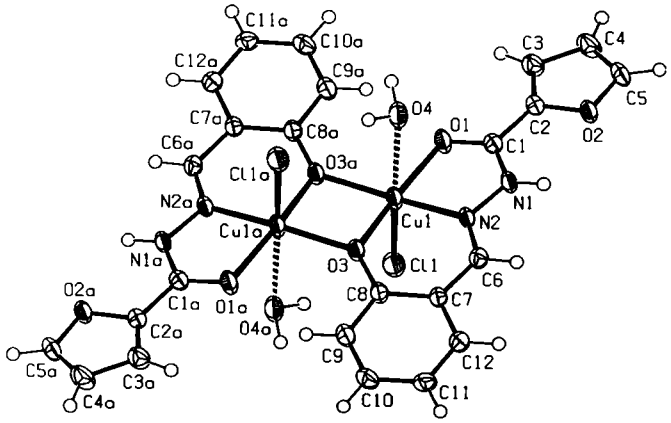
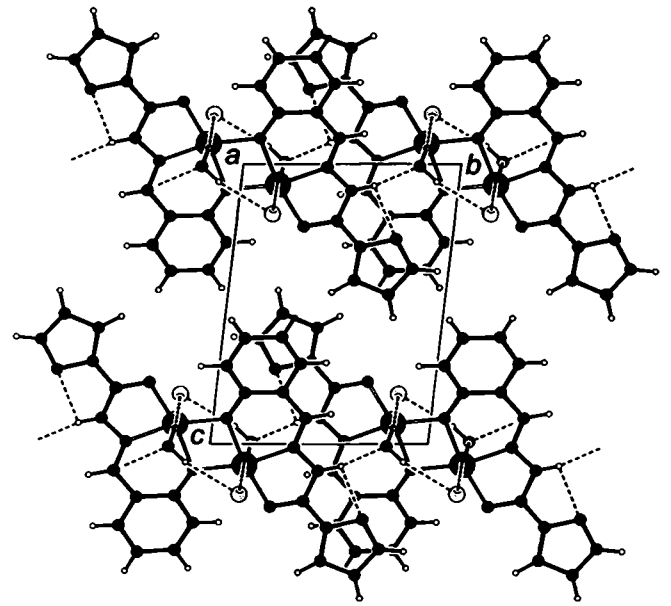
Crystal structure of *trans*-diaqua-dichloro-bis(μ-salicylaldehyde-2-furanoylhydrazonato)dicopper(II), $Cu_2[(C_4H_3O)CONHNCH(C_6H_4O)]_2Cl_2(H_2O)_2$

P. M. Haba[I], O. Diouf[I], A. Sy[I], M. L. Gaye*[I], A. S. Sall[I], A. H. Barry[II] and T. Jouini[III]

[I] Université Cheikh Anta Diop, Département de Chimie, Faculté des Sciences et Techniques, Dakar, Senegal
[II] Université de Nouakchott, Faculté des Sciences, Département de Chimie, Nouakchott, Mauritania
[III] Université de Tunis, Faculté des Sciences, Département de Chimie, Campus universitaire, 1060 Tunis, Tunisia

Received August 2, 2005, accepted and available on-line September 27, 2005; CCDC no. 1267/1619





Abstract

$C_{12}H_{11}ClCuN_2O_4$, triclinic, $P\bar{1}$ (no. 2), a = 7.4627(3) Å, b = 8.8894(3) Å, c = 11.0866(4) Å, α = 89.251(1)°, β = 73.545(1)°, γ = 65.975(2)°, V = 639.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.041, $wR_{ref}(F^2)$ = 0.124, T = 173 K.

Source of material

Salicylaldehyde (10 mmol) was added to a solution of furanoylhydrazide (10 mmol) in 20 ml of ethanol. The mixture was heated under reflux for two hours. The resulting solution was left standing for one day. The white powder was isolated by filtration and washed with ether before dried under vacuum to obtain salicylaldehyde hydrazone (H_2L). An ethanol solution of $CuCl_2 \cdot 2H_2O$ (1 mmol) was then added to an ethanol solution of ligand H_2L (1 mmol). The resulting green solution was stirred and refluxed for three hours. After the filtrate had stood for two days in air, at room temperature a green precipitate was obtained. Crystals of $[Cu_2(H_2O)_2(Cl)_2(HL)_2]$ suitable for X-ray analysis were obtained by slow evaporation from an ethanolic solution.

Discussion

The structure analysis revealed a dinuclear centrosymmetric complex (figure, top) being similar with two other related dinuclear salicylaldehyde-hydrazonato Cu(II) complexes, but different to the mononuclear aqua-chloro-(salicylaldehyde acetyl-hydrazonato)-copper(II) complex [1]. One Cu(II) atom has a distorted square pyramidal coordination geometry. Deviation from 90° of the bond angles involving the chelation is observed (∠N2–Cu–O1 = 80.72(8)°, ∠O3–Cu–O1^i = 106.51(7)°, ∠N2–Cu–O3 = 90.70(8)°, ∠O3–Cu–O3^i = 79.74(8)°) presumably due to the formation of five-membered ring [2]. The apical position is occupied by the chloride ion with a distance of 2.6002(7) Å. Relevant bonds distances are: d(N2—Cu) = 1.948(2) Å, d(O1—Cu) = 2.009(2) Å, d(O3—Cu) = 1.954(2) Å and d(O3^i—Cu) = 1.977(2) Å. The aqua molecule completes an elongated octahedron with a Cu—O4 distance of 2.830(2) Å. The C3=O2 and C6=N1 distances of 1.258(3) Å and 1.229(4) Å, respectively, show essentially double-bond character. The intermetallic distance Cu1···Cu1^i is 3.0168(6) Å (symmetry code: (i) $-x,2-y,2-z$). Layers parallel (100) are formed by O–H···Cl and N–H···O bonds (figure, bottom).

Table 1. Data collection and handling.

Crystal:	green prism, size 0.10 × 0.10 × 0.10 mm
Wavelength:	Mo K_α radiation (0.71070 Å)
μ:	19.28 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	60.08°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7413, 3736
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2971
$N(param)_{refined}$:	181
Programs:	SHELXS-86 [3], SHELXL-97 [4], ORTEP-II [5], WinGX [6], PLATON [7]

* Correspondence author (e-mail: mlgayeastou@yahoo.fr)

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.1487	0.5255	0.5426	0.038
H(2)	2*i*	–0.2507	0.7708	1.5493	0.044
H(3)	2*i*	–0.3235	0.9481	1.3831	0.040
H(4)	2*i*	0.2485	0.3873	0.9221	0.030
H(5)	2*i*	0.2864	0.4619	1.1081	0.030
H(6)	2*i*	0.0914	1.0822	1.2779	0.039
H(7)	2*i*	0.7851	0.0324	0.5575	0.039
H(8)	2*i*	0.6530	0.3197	0.5497	0.035
H(9)	2*i*	0.6467	0.4893	0.7090	0.033
H(10)	2*i*	0.3602	0.1031	0.9360	0.050
H(11)	2*i*	0.3339	0.1681	1.0572	0.050

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)	2*i*	0.10667(5)	0.82946(3)	0.92671(3)	0.0454(2)	0.0141(2)	0.0247(2)	–0.0099(1)	–0.0173(1)	0.0020(1)
Cl(1)	2*i*	0.4602(1)	0.83891(8)	0.82173(7)	0.0414(4)	0.0278(3)	0.0364(4)	–0.0144(3)	–0.0132(3)	0.0029(3)
O(1)	2*i*	0.1295(3)	0.6901(2)	0.7773(2)	0.052(1)	0.0163(8)	0.0267(9)	–0.0122(8)	–0.0188(8)	0.0020(7)
O(2)	2*i*	–0.2573(3)	0.7408(2)	1.2687(2)	0.040(1)	0.0175(8)	0.0265(9)	–0.0113(7)	–0.0132(8)	0.0001(7)
O(3)	2*i*	0.0603(3)	0.9379(2)	1.0919(2)	0.042(1)	0.0154(8)	0.0237(9)	–0.0083(7)	–0.0170(7)	0.0011(7)
O(4)	2*i*	0.2783(3)	0.1891(2)	0.9918(2)	0.055(1)	0.0201(9)	0.038(1)	–0.0101(8)	–0.0222(9)	0.0029(8)
N(1)	2*i*	0.2151(3)	0.4906(2)	0.9055(2)	0.037(1)	0.0145(9)	0.025(1)	–0.0104(8)	–0.0133(8)	0.0014(8)
N(2)	2*i*	0.1969(3)	0.6133(2)	0.9888(2)	0.031(1)	0.0142(9)	0.025(1)	–0.0085(8)	–0.0107(8)	–0.0005(7)
C(1)	2*i*	0.1779(4)	0.5410(3)	0.7975(2)	0.028(1)	0.021(1)	0.024(1)	–0.0104(9)	–0.0091(9)	0.0009(9)
C(2)	2*i*	0.1982(4)	0.4196(3)	0.7034(2)	0.029(1)	0.019(1)	0.027(1)	–0.0097(9)	–0.011(1)	0.0008(9)
C(3)	2*i*	0.1868(4)	0.4286(3)	0.5843(3)	0.045(2)	0.028(1)	0.027(1)	–0.017(1)	–0.018(1)	0.004(1)
C(4)	2*i*	–0.2430(5)	0.7359(4)	1.4666(3)	0.050(2)	0.036(2)	0.030(1)	–0.021(1)	–0.015(1)	–0.004(1)
C(5)	2*i*	–0.2827(4)	0.8317(3)	1.3750(3)	0.044(2)	0.022(1)	0.036(2)	–0.015(1)	–0.014(1)	–0.005(1)
C(6)	2*i*	0.2415(4)	0.5748(3)	1.0928(2)	0.030(1)	0.019(1)	0.027(1)	–0.0101(9)	–0.012(1)	0.0055(9)
C(7)	2*i*	0.2270(4)	0.6925(3)	1.1861(2)	0.027(1)	0.018(1)	0.024(1)	–0.0092(9)	–0.0100(9)	0.0027(9)
C(8)	2*i*	0.1414(4)	0.8673(3)	1.1828(2)	0.029(1)	0.018(1)	0.024(1)	–0.0095(9)	–0.0090(9)	0.0022(9)
C(9)	2*i*	0.1429(4)	0.9655(3)	1.2796(3)	0.047(2)	0.021(1)	0.036(2)	–0.014(1)	–0.022(1)	0.002(1)
C(10)	2*i*	0.7825(4)	0.1028(3)	0.6224(3)	0.045(2)	0.030(1)	0.031(1)	–0.017(1)	–0.020(1)	–0.000(1)
C(11)	2*i*	0.7035(4)	0.2733(3)	0.6174(3)	0.034(1)	0.031(1)	0.028(1)	–0.015(1)	–0.018(1)	0.008(1)
C(12)	2*i*	0.7000(4)	0.3728(3)	0.7121(2)	0.034(1)	0.023(1)	0.028(1)	–0.012(1)	–0.015(1)	0.006(1)

Acknowledgments. The authors thank the Agence Universitaire de la Francophonie for his financial support (AUF-PSCI no. 6301PS48) and Mr. Sada Ndiaye Dage of the Ministry of National Education of Senegal for material support.

References

1. Ainscough, E. W.; Brodie, A. M.; Dobbs, A. J.; Ranford, J. D.; Waters, J. M.: Antitumour copper(II) salicylaldehyde benzoylhydrazone (H_2sb) complexes: physicochemical properties and the single-crystal X-ray structures of $[\{Cu(H_2sb)(CCl_3CO_2)_2\}_2]$ and $[\{Cu(Hsb)(ClO_4)(C_2H_5OH)\}_2]$ and the related salicylaldehyde acetylhydrazone (H_2sa) complex, $[Cu(Hsa)Cl(H_2O)]\cdot H_2O$. Inorg. Chim. Acta **267** (1998) 27-38.
2. Mao, H.-Y.; Shen, X.-Q.; Li, G.; Zhang, H.-Y.; Xu, C.; Liu, H.-L.; Wang, E.-B.; Wu, Q.-A.; Hu, H.-W.; Zhu, Y.: Novel hydrogen-bonded and π-π interaction networks generated from reaction of copper(II) chloride hydrates with heterocyclic diimines (2,2′-bipyridine, 1,10-phenantroline) and bidentate diamine (ethylenediamine). Polyhedron **23** (2004) 1961-1967.
3. Sheldrick, G. M.: SHELXS-86. A direct methods program for crystal structure determination. In: *Crystallographic Computing 3* (Eds. G. M. Sheldrick, C. Kruger, R. Goddard), pp. 175-189. Oxford University Press, UK 1985.
4. Sheldrick, G. M. SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
5. Johnson, C. K.: ORTEP-II. A Fortran Thermal-Ellipsoid Plot Program. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA 1976.
6. Farrugia, L. J.: WinGX suite for small-molecule single-crystal crystallography. J. Appl. Crystallogr. **32** (1999) 837-838.
7. Spek, A. L.: Single-crystal structure validation with the program PLATON. J. Appl. Crystallogr. **36** (2003) 7-13.