

Polyhedron 21 (2002) 1761-1766



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# The structural chemistry of copper(II) hinokitiol and its adducts

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Received 25 March 2002; accepted 15 May 2002

## Abstract

The structure of copper(II) hinokitiol has been determined. Two modifications are possible, one containing only isolated, fourcoordinate, square-planar complexes, while a second modification contains both monomers, along with eclipsed and staggered dimers generated through intermolecular  $O \rightarrow Cu$  interactions. Five-coordinate copper is also present in the adduct Cu(hino)<sub>2</sub>. pyridine, whose structure has also been determined for comparison. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Copper; Hinokitiol; X-ray; Adduct

## 1. Introduction

There is both biological and medicinal interest in compounds which have ketone and hydroxyl functional groups adjacent to one another and which exhibit antibacterial or -fungal properties [1-6]. Two compounds which fit into this category are 2-hydroxy-2,4,6-cycloheptatrien-1-one (tropolone I) and 6-isopropyltropolone (II), commonly called hinokitiol [7].



Tropolone is a representative of a family of nonbenzenoid aromatic compounds and has been studied in depth over the last 40 years. The functional groups of tropolone allow complexation of a number of different

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metal ions  $(M^{2+}, M^{3+} \text{ and } M^{4+})$  [8–13]. Hinokitiol (Hhino) is isolated from the wood of the *Chamacyparis taiwanesis* tree, and since its discovery in 1936 [14] it has been reported to exhibit anti-microbial effects [7] and also to stimulate plant growth. However, in contrast to metal tropolonates, analogous metal derivatives of hinokitiol remain largely unexplored, though we have recently reported the structures of X<sub>2</sub>Sn(hino)<sub>2</sub> (X = F, Cl) [15] and Zn(hino)<sub>2</sub>·EtOH [16]. The structure of In(hino)<sub>3</sub> has also been determined [17].

Our general interest in this area lies in the inorganic chemistry of M(II) (M = Cu, Zn, Sn) derivatives of orally viable  $\alpha$ -hydroxyketones e.g. maltol, ethylmaltol as well as hinokitiol [15,16], which have relevance to novel dental formulations [18]. In this paper, we report on the unusual structural chemistry of Cu(II) hinokitiol and its adduct chemistry.

## 2. Experimental

Infrared spectra (cm<sup>-1</sup>) were recorded as Nujol mulls between NaCl plates using a Nicolet 510P FT-IR spectrophotometer, and elemental analyses were performed using an Carlo–Erba Strumentazione E.A. model 1106 microanalyser operating at 500 °C. Starting materials were commercially obtained and used without further purification.

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#### 2.1. Synthesis of Cu(II) hinokitiol (1)

Hinokitiol (0.30 g, 1.83 mmol) was dissolved in aq. EtOH (50:50, 50 ml) with stirring. Once dissolved, a solution of copper acetate (0.19 g, 0.95 mmol) was added and the stirring continued. After stirring and gentle heating for 2 h, the green/blue solution was allowed to stand for 1 week after which time the product crystallised from solution as green/blue plate-like crystals. *Anal*. Found (Calc. for  $C_{20}H_{22}CuO_4$ ): C, 61.6 (61.6); H, 5.64 (5.69)%. Selected IR data (cm<sup>-1</sup>): 1600, 1574 v(C=O); 1512 v(C=C); 1286–1228 v(C–O).

#### 2.2. Synthesis of $(hinokitiol)_2Cu \cdot (pyridine)$ (2)

Pyridine (5 ml) was added to a solution of **1** (0.30 g, 0.77 mmol) in C<sub>6</sub>H<sub>5</sub>CH<sub>3</sub> (30 ml). After stirring for 20 min, the solution was cooled to -15 °C whereupon a green solid appeared (0.06 g; 17%). Some crystals suitable for X-ray crystallography were obtained from the filtrate. *Anal*. Found (Calc. for C<sub>25</sub>H<sub>27</sub>CuNO<sub>4</sub>): C, 63.7 (64.0); H, 5.79 (5.76); N, 2.85 (2.98)%. Selected IR data (cm<sup>-1</sup>): 1583, 1569, 1496 v(C=O) and v(C=C); 1261, 1231 v(C-O).

## 2.3. Synthesis of $(hinokitiol)_2Cu \cdot (bipyridine)$ (3)

Bipyridine (1.00 g, 6.25 mmol) was dissolved in  $C_6H_5CH_3$  (30 ml) and then added to a solution of 1 (0.40 g, 1.03 mmol) in  $C_6H_5CH_3$  (50 ml). The mixture was stirred for 20 min and then left at room temperature for few days, yielding a brown solid (0.44 g, 78%). *Anal*. Found (Calc. for  $C_{30}H_{30}CuN_2O_4$ ): C, 65.5 (65.9); H, 5.48 (5.49); N, 5.83 (5.13)%. Selected IR data (cm<sup>-1</sup>): 1594, 1554, 1511 v(C=O) and v(C=C); 1278, 1199 v(C-O).

#### 2.4. Crystallography

Suitable crystals of **1** were obtained either from EtOH (modification 1) or from  $C_6H_5CH_3$  in the presence of urea (modification 2). Crystals of **2** were obtained from a dilute solution in  $C_6H_5CH_3$  (Table 1).

For modification 1 of 1, crystallographic measurements were made at 293(2) K on a CAD4 automatic four-circle diffractometer in the range  $2.40^{\circ} < \theta < 23.92^{\circ}$ . Data (1685 reflections) were corrected for Lp and 10% decay of the crystal in the X-ray beam. An absorption correction was not applied. In the final least-squares cycles all atoms were allowed to vibrate anisotropically. Hydrogen atoms were included at calculated positions where relevant except for H3, H4, H5 and H7 which were easily located in the penultimate difference Fourier map and refined at a distance of 0.93 Å from the carbon atoms to which they are attached.

For modification 2 of 1, crystallographic measurements were made at 150 K on a Nonius Kappa CCD diffractometer in the range  $3.80^{\circ} < \theta < 27.48^{\circ}$ . Data (54148 reflections) were corrected for Lp, extinction and absorption (max. and min. transmission factors: 1.030 and 0.974, respectively). The asymmetric unit consisted of 2.5 independent molecules. The two full molecules are located proximate to inversion centres and hence dimerise with nearby lattice neighbours. The central copper atom [Cu(3)] in the remaining independent half-molecule is seated on an inversion centre which serves to generate the full molecule in this case. Disorder in one of the isopropyl groups [C(39)/C(40)] was successfully modelled as a 55:45 occupancy ratio. In the final least-squares cycles, all atoms were allowed to vibrate anisotropically and hydrogen atoms were included at calculated positions throughout.

Crystallographic measurements for **2** were made at 293(2) K on a CAD4 automatic four-circle diffractometer in the range  $2.24^{\circ} < \theta < 24.98^{\circ}$ . Data (4337 reflections) were corrected for Lp but not for absorption. In the final least-squares cycles, all atoms were allowed to vibrate anisotropically. Hydrogen atoms were included at calculated positions where relevant.

In all three cases, refinement was based on  $F_0^2$ . Software used: SHELXS-86 [19], SHELXL-97 [20], ORTEX [21]. The asymmetric units of 1 (two modifications) and 2 are displayed in Figs. 1–3 along with the atomic numbering scheme. Selected metric data for modification 1 of 1 and 2 are given in the figure captions, while that for modification 2 of 1 are given in Table 2.

## 3. Results and discussion

Cu(II) hinokitiol (1) was synthesised using the method detailed in Eq. (1). After 1 week, a large number of blue plate-like crystals had formed in the solution. Micro-analysis of these crystals showed the complex to be anhydrous.



We have previously noted that copper derivatives of  $\alpha$ -hydroxyketones (maltol, hinokitiol etc.) in which the copper is four-coordinate, square-planar geometry are notably anti-bacterially and/or -microbially active, whereas the analogous zinc species, in which the zinc is either five- or six-coordinate, are markedly less so [18].

Table 1 Crystallographic data for **1** and **2** 

	1 (modification 1)	1 (modification 2)	2
Empirical formula	C <sub>20</sub> H <sub>22</sub> CuO <sub>4</sub>	C <sub>20</sub> H <sub>22</sub> CuO <sub>4</sub>	C <sub>25</sub> H <sub>27</sub> CuNO <sub>4</sub>
Formula weight	389.92	389.92	469.02
Temperature (K)	293(2)	150(2)	293(2)
$\lambda$ (Mo K $\alpha$ ) (Å)	0.71069	0.71069	0.71069
Crystal system	monoclinic	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/n$	$P2_1/n$
a (Å)	9.254(2)	16.7420(2)	8.616(2)
b (Å)	9.997(2)	15.5370(2)	26.848(4)
c (Å)	11.124(3)	18.3010(3)	9.910(2)
β(°)	113.86(2)	99.4000(6)	102.56(2)
V (Å <sup>3</sup> )	941.2(4)	4696.54(11)	2237.5(8)
Ζ	2	10	4
$\rho_{\rm calc}$ (Mg m <sup>-3</sup> )	1.376	1.379	1.392
$\mu  ({\rm mm}^{-1})$	1.180	1.183	1.007
Crystal size (mm)	0.3  imes 0.3  imes 0.5	0.25  imes 0.10  imes 0.10	0.25  imes 0.20  imes 0.20
$\theta$ Range (°)	2.40-23.92	3.80-27.48	2.24-24.98
Reflections collected	1685	54 148	4337
Independent reflections	1475 $[R_{int} = 0.0396]$	$10712 [R_{int} = 0.0471]$	$3928 [R_{int} = 0.0326]$
Final <i>R</i> indices $[I > 2\sigma(I)]$	$R_1 = 0.0430, wR_2 = 0.0704$	$R_1 = 0.0384, wR_2 = 0.1000$	$R_1 = 0.0441, wR_2 = 0.1232$
R indices (all data)	$R_1 = 0.1066, w R_2 = 0.0921$	$R_1 = 0.0558, wR_2 = 0.1139$	$R_1 = 0.1271, wR_2 = 0.1681$

In order to assess the importance of metal coordination number on activity, we have prepared complexes of 1 by reaction with N-donor Lewis bases (Eq. (2)). 1:1 adducts with pyridine (2) and 2,2'-bipyridine (3) have been isolated, which presumably contain five- or six-coordinate copper, respectively.



## 3.1. The structure of Cu(II) hinokitiol (1)

Cu(II) hinokitiol, prepared as in Eq. (1), was recrystallised from aqueous ethanol, yielding a crystal of approximate dimensions  $0.3 \times 0.3 \times 0.5$  mm which was used for data collection. Selected structural data are included in the caption to Fig. 1. This structure will be discussed as modification 1. An attempt to prepare an adduct of 1 with urea in toluene failed, and green crystals of 1 were recovered but whose cell parameters [a = 16.7420(2); b = 15.5370(2); c = 18.3010(3) Å;  $\beta =$ 99.4000(6)°] were different to those of modification 1 [a = 9.254(2); b = 9.997(2); c = 11.124(3) Å;  $\beta =$ 113.86(2)°]. This second crystal structure will be dis-



Fig. 1. The asymmetric unit of modification 1 of compound **1** showing the labelling scheme used in the text. Thermal ellipsoids are at the 30% probability level. Selected metrical data: Cu(1)–O(1) 1.904(3), Cu(1)–O(2) 1.900(2), O(1)–C(2) 1.293(5), O(2)–C(1) 1.296(5) Å; O(1)–Cu(1)–O(1') 180.0, O(2)–Cu(1)–O(2') 180.0, O(1)–Cu(1)–O(2) 83.84(13), O(1)–Cu(1)–O(2') 96.16(13), C(2)–O(1)–Cu(1) 113.5(3), C(1)–O(2)–Cu(1) 113.5(3)°. Symmetry related atoms are generated using -x, -y, -z.

cussed as modification 2. Both forms adopt monoclinic  $P2_1/c$  (or  $P2_1/n$ ) symmetries.

In modification 1 (Fig. 1), the asymmetric unit comprises half of a molecule, the remaining portion being generated via an inversion centre at the origin on which the copper is sited. The Cu(II) is four-coordinate and in a square-planar environment. The molecule as a whole is essentially planar [maximum deviation from mean plane: 0.029 Å for O(2)]. The Cu-O bonds are equivalent [Cu-O(1), 1.904(3); Cu-O(2), 1.900(2) Å] and the  $\langle O(1)-Cu-O(2) \rangle$  bond angle is  $83.8(1)^{\circ}$ . The Cu–O bond lengths are in agreement with other Cu(II) complexes, e.g. Cu(II) erythritol [Cu-O: 1.912(4), 1.922(4) Å] [22]. Both of the C-O bond lengths in 1 compare well with those found in a number of other metal tropolonates, i.e. 1.27-1.30 Å and Zn(II) hinokitiol [1.277(2)-1.306(2) Å] [16]. Furthermore, upon coordination to the Cu(II) the C-O bond lengths become equal [1.293(5), 1.296(3) Å], indicating a similar bond order in both. This is a more symmetric arrangement than the equivalent bonds in hinokitiol itself (C-O: 1.349; C=O: 1.261 Å) [23]. The lengths of the C–C bonds in the hinokitiol ring of 1 are approximately equal (approximately 1.38 Å) with one notable exception. The C(1)-C(2) bond is significantly longer than the others in the ring, indicating that it is not involved to the same extent in the  $\pi$ -electron delocalisation within the C<sub>7</sub> unit. The value of 1.464(6) Å observed for the C(1)-C(2)bond in 1 is closer to that expected for a  $C(sp^3)-C(sp^3)$  single bond. For comparison, the  $\pi$ -electron system in hinokitiol is also partially delocalised, though the formal double bonds [C(1)–C(7), C(6)–C(5) and C(4)–C(3); 1.367, 1.355 and 1.364 Å, respectively] are more clearly distinguishable from the formal single bonds [C(7)–C(6), C(5)–C(4) and C(3)–C(2); 1.422, 1.413 and 1.413 Å, respectively] than in **1**. The C(1)–C(2) bond in the ligand (1.469 Å) is, however, significantly longer than the other carbon–carbon bonds and is comparable with that in **1** [23].

In modification 2 of 1, obtained from toluene in an attempt to prepare a urea adduct, there are two and half molecules in the asymmetric unit; two molecules are in general positions, while half molecule has the copper atom seated on an inversion centre (Fig. 2), as with modification 1. For each molecule, the copper is nominally four-coordinate and in a square-planar environment. The molecule generated by the inversion centre [molecule A, containing Cu(3)] has the isopropyl groups adopting a trans configuration and this is also the case for one of the two other molecules [molecule B containing Cu(2)]; in this respect, both molecules replicate the structure of modification 1. However, in the remaining molecule [molecule C containing Cu(1)] the isopropyl groups are cis to each other. This is unusual, as for previous structures of  $\alpha$ -hydroxyketones, the alkyl groups are *trans* to each other e.g.  $Zn(hino)_2$ . EtOH [16], Zn(maltol)<sub>2</sub> [24], and 2 (see below).



Fig. 2. The asymmetric unit of modification 2 of compound 1 showing the labelling scheme used in the text. Thermal ellipsoids are at the 30% probability level.



Fig. 3. The asymmetric unit of **2** showing the labelling scheme used in the text. Thermal ellipsoids are at the 30% probability level. Selected metrical data: Cu(1)-O(1) 1.940(3), Cu(1)-O(2) 1.945(3), Cu(1)-O(3) 1.965(3), Cu(1)-O(4) 1.933(3), Cu(1)-N(1) 2.311(5), O(1)-C(1) 1.287(5), O(2)-C(2) 1.278(6), O(3)-C(8) 1.287(5), O(4)-C(9) 1.277(6) Å; O(1)-Cu(1)-O(2) 82.73(14), O(1)-Cu(1)-O(3) 97.28(14), O(1)-Cu(1)-O(4) 172.2(2), O(2)-Cu(1)-O(3) 155.2(2), O(2)-Cu(1)-O(4) 94.3(2), O(3)-Cu(1)-O(4) 82.34(14), O(1)-Cu(1)-N(1) 92.3(2), O(2)-Cu(1)-N(1) 99.0(2), O(3)-Cu(1)-N(1) 105.8(2),  $O(4)-Cu(1)-N(1) 95.3(2)^{\circ}$ .

The two full molecules in modification 2 (B and C) are proximate to inversion centres and hence dimerise with nearby lattice neighbours (Fig. 2). In the dimer based on

Table 2 Selected bond lengths (Å) and angles (°) for modification 2 of 1  $^{\rm a}$ 

Molecule A	Molecule B	Molecule C
1.910(2)		
1.901(2)		
		1.919(2)
		1.920(2)
		1.932(2)
		1.933(2)
	1.915(2)	
	1.921(2)	
	1.939(2)	
	1.922(2)	
		2.476(2)
	2.511(2)	
84.53(8)	84.27(7)	83.86(7)
95.47(8)	97.95(7)	99.15(7)
180.0	174.64(7)	177.40(7)
180.0	166.88(7)	165.00(8)
95.47(8)	93.36(7)	93.55(7)
84.53(8)	83.29(7)	83.26(7)
	Molecule A 1.910(2) 1.901(2) 84.53(8) 95.47(8) 180.0 180.0 95.47(8) 84.53(8)	Molecule A  Molecule B    1.910(2)

<sup>a</sup> Symmetry transformation used to generate equivalent atoms: -x, -y, -z.

<sup>b</sup> Atomic labels refer to molecule C; for molecules A [Cu(3)] and B [Cu(2)], oxygens are O(9, 10) and O(5–8), respectively.

B, the isopropyl groups sit above each other (eclipsed conformation), while in the dimer of C, they are on opposite sides of the stacked seven-membered rings (staggered conformation). Thus, molecule B minimises repulsions between isopropyl groups within the monomer but maximises it between molecules on dimer formation, while the reverse is true for molecule C.

In the non-dimerised molecule A, the Cu-O bond lengths [1.901(2), 1.909(2) Å] are similar than those observed in modification 1 [1.900(2), 1.904(3) Å]. For dimers based on molecules B and C, the intramolecular Cu–O bond lengths [1.915(2)-1.939(2) Å] are longer than those in modification 1 and thus the effect of the dimerisation is similar to the effect of the pyridine coordination, as in 2 (see below). The intermolecular interactions [Cu(1)-O(4):2.476(2); Cu(2) - O(8): 2.511(2) Å] are clearly longer and weaker than the analogous intramolecular bonds, with the eclipsed dimerisation in molecule B manifesting itself in the longer of these two contacts. The intermolecular interactions in both cases do, however, generate almost perfect  $Cu_2O_2$ squares [ < Cu(1) - O(4) - Cu(1'): $93.09(6)^{\circ}$ ; < Cu(2)–O(8)–Cu(2'): 94.80(6)°] at the heart of each dimer.

The coordination chemistry of **1** is thus exceedingly flexible, as both *cis* and *trans* ligand arrangements are possible, as are both monomers and dimers.

### 3.2. The structure of Cu(II) hinokitiol pyridine adduct 2

Compound 2 crystallises in the monoclinic space group  $P2_1/n$ . The copper adopts a square pyramidal geometry with the pyridine in the axial position (Fig. 3),

the copper lies 0.125 Å above the plane of the four oxygen atoms. The Cu–O bond lengths [1.940(3), 1.933(3), 1.945(3), 1.985(3) Å] are longer than those observed in isolated molecules of **1** [1.900(2)–1.909(2) Å]. The structure is similar to the one obtained for Cu(acrp)<sub>2</sub>·pyridine (Hacrp: 4-hydroxy-6-methyl-3-[3-dimethylaminoacryloyl]-2*H*-pyran-2-one), and the Cu–N bond [2.311(5) Å] is equivalent with the Cu–N bond of this species [2.310(10) Å] [25].

The electronic spectra of Ni(II) and Cu(II) species incorporating both amines and hinokitiol i.e.  $[M(hino)(diamine)]^+$  have been studied [26,27] and such compounds exploited in the extraction of unprotected amino acids [26].

## 4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 181131–181133 for compounds 1 (two modifications) and 2. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ ccdc.cam.uk or www: http://www.ccdc.cam.ac.uk).

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