metal-organic compounds

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Bis(2-methoxyphenolato- $\kappa^2 O, O'$)copper(II)

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Key indicators: single-crystal X-ray study; T = 293 K; mean σ (C–C) = 0.007 Å; R factor = 0.051; wR factor = 0.129; data-to-parameter ratio = 14.4.

In the title compound, $[Cu(C_7H_7O_2)_2]$, the asymmetric unit contains one and a half molecules with the central Cu(II) atoms situated on a general position and on a centre of inversion, respectively. Both Cu(II) atoms show a similar slightly distorted square-planar coordination, resulting from four O atoms of two 2-methoxyphenolate anions.

Related literature

For 2-methoxy-phenol compounds, see: Campello *et al.* (1997); Floriani *et al.* (1988); Minhas *et al.* (1993); Kuo *et al.* (1999); Schumann *et al.* (1996); Sobota *et al.* (2001).



Experimental

Crystal data [Cu(C₇H₇O₂)₂]

 $M_r = 333.82$

Triclinic, P1
a = 9.5190 (19) Å
b = 11.540 (2) Å
c = 12.488 (3) Å
$\alpha = 102.83 \ (3)^{\circ}$
$\beta = 103.93 \ (3)^{\circ}$
$\gamma = 111.20 \ (3)^{\circ}$

Data collection

289 parameters

Bruker P4 diffractometer Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $T_{\min} = 0.656, T_{\max} = 0.857$

Refinement $R[F^2 > 2\sigma(F^2)] = 0.051$ 4 restraints $wR(F^2) = 0.129$ H-atom parameters constrainedS = 0.93 $\Delta \rho_{max} = 0.70 \text{ e } \text{\AA}^{-3}$ 4164 reflections $\Delta \rho_{min} = -0.63 \text{ e } \text{\AA}^{-3}$

Data collection: *XSCANS* (Bruker, 1997); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

V = 1166.7 (6) Å³

Mo $K\alpha$ radiation

 $0.23 \times 0.12 \times 0.08 \text{ mm}$

6930 measured reflections 4164 independent reflections

3466 reflections with $I > 2\sigma(I)$

 $\mu = 1.42 \text{ mm}^-$ T = 293 K

 $R_{\rm int} = 0.037$

7 - 3

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2068).

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supporting information

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Bis(2-methoxyphenolato- $\kappa^2 O, O'$)copper(II)

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S1. Comment

2-Methoxy-phenol ligand can act as either mondentate ligand (Campello, *et al.*, 1997), or didentate ligand (Sobota, *et al.*, 2001), or mu_2 -o ligand or mu_3 :eta¹:eta²-O ligand (Schumann, *et al.* 1996) or mu_4 :eta¹:eta³-O ligand (Floriani, *et al.* 1988). However, copper compound with 2-Methoxy-phenol have not been reported till today (http://www.ccdc.cam.ac.uk/). The title compound, (I), is a new Cu^{II} complex prepared by reaction of 2-Methoxy-phenol and Copper(II) nitrate using solvothermal technique.

There are one CuÎl[^] atom and two *L*⁻ ligand in the asymetric unit. The Cu^{II} atom has a slightly distorted square-planar environment, formed by four O atoms from two different *L*⁻ ligands. The *L*⁻ ligand binds to copper in a didentate mode, through two O atoms. In the title complex, the two copper lied in the different position that the Cu2 is at the center of symmetry (010) plane and the Cu1 is at a general position (Fig. 2). The complex further constructed a 3-D network through very weak C–H···O hydrogen bond (C21–H21···O1ⁱ, 3.426 (1) Å, symmetry code: (i) 1 - *y*, 2 - *y*, 1 - *z*) and C–H···p hydrogen bond (C16···Pⁱⁱ, 3.652 (1) Å, symmetry code: (ii) 1 + *x*, *y*, *z*).

S2. Experimental

A solution of (0.124 g, 1 mmol) 2-Methoxy-phenol and (0.056 g, 1 mmol) potassium hydroxide in 8 ml absolute methanol was added ((0.125 g, 0.5 mmol) Copper nitrate tetrahydrate. The solution was placed in a 15-ml Tetlon-lined stainless steel parr bomb. The bomd was heated at 363 k for 96 h. The cooled mixture yielded blue block-shaped crystal of (1) in about 71% yield. The crystals were washed with methanol and then dried in air.

S3. Refinement

H atoms were positioned geometrically and refined with a riding model, with distances 0.96 Å(CH₃) or 0.93 Å(aromatic ring). and with $U_{iso}(H) = 1.2 U_{eq}(aromatic ring)$ or $U_{iso}(H) = 1.5 U_{eq}(CH_3)$.



Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids. Symmetry codes: (A) -x + 1, -y + 2, -z + 1.



Figure 2

Packing diagram of title complex, hydrogen atoms were omitted.

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Crystal data

 $[Cu(C_7H_7O_2)_2]$ $M_r = 333.82$ Triclinic, $P\overline{1}$ Hall symbol: -P 1 a = 9.5190 (19) Åb = 11.540(2) Å c = 12.488 (3) Å $\alpha = 102.83 (3)^{\circ}$ $\beta = 103.93 (3)^{\circ}$ $\gamma = 111.20 (3)^{\circ}$ V = 1166.7 (6) Å³

Data collection

Bruker P4
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)
$T_{\min} = 0.656, \ T_{\max} = 0.857$

Refinement	
Refinement on F^2	Secondary atom site location: difference Fourier
Least-squares matrix: full	map
$R[F^2 > 2\sigma(F^2)] = 0.051$	Hydrogen site location: inferred from
$wR(F^2) = 0.129$	neighbouring sites
S = 0.93	H-atom parameters constrained
4164 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2 + 1.5P]$
289 parameters	where $P = (F_{o}^{2} + 2F_{c}^{2})/3$
4 restraints	$(\Delta/\sigma)_{\rm max} = 0.001$
Primary atom site location: structure-invariant	$\Delta \rho_{\rm max} = 0.70 \ { m e} \ { m \AA}^{-3}$
direct methods	$\Delta \rho_{\rm min} = -0.63 \ {\rm e} \ {\rm \AA}^{-3}$

Z = 3

F(000) = 513

 $\theta = 3.1 - 25.3^{\circ}$

 $\mu = 1.42 \text{ mm}^{-1}$

T = 293 K

Block, blue

 $R_{\rm int} = 0.037$

 $h = -11 \rightarrow 11$ $k = -13 \rightarrow 13$ $l = -14 \rightarrow 12$

 $D_{\rm x} = 1.425 {\rm Mg} {\rm m}^{-3}$

 $0.23 \times 0.12 \times 0.08 \text{ mm}$

6930 measured reflections 4164 independent reflections 3466 reflections with $I > 2\sigma(I)$

 $\theta_{\text{max}} = 25.3^{\circ}, \ \theta_{\text{min}} = 3.1^{\circ}$

Mo *K* α radiation, $\lambda = 0.71073$ Å

Cell parameters from 4216 reflections

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes. **Refinement**. Refinement of F^2 against ALL reflections. The weighted *R*-factor w*R* and goodness of fit *S* are based on F^2 , conventional R-factors R are based on F, with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (A^2)

	x	у	Ζ	$U_{ m iso}$ */ $U_{ m eq}$
Cu1	0.52201 (5)	0.70662 (4)	0.76824 (4)	0.03605 (16)
Cu2	0.5000	1.0000	0.5000	0.03477 (19)
C1	0.1962 (4)	0.5274 (4)	0.7391 (3)	0.0393 (9)

C2	0.0931 (5)	0.4117 (4)	0.7551 (4)	0.0544 (11)
H2	0.1388	0.3665	0.7934	0.065*
C3	-0.0719 (6)	0.3655 (5)	0.7154 (5)	0.0700 (14)
H3	-0.1355	0.2901	0.7273	0.084*
C4	-0.1437 (6)	0.4304 (6)	0.6579 (5)	0.0746 (15)
H4	-0.2552	0.3979	0.6298	0.090*
C5	-0.0486(5)	0.5435 (5)	0.6426 (4)	0.0628 (13)
Н5	-0.0982	0.5867	0.6046	0.075*
C6	0.1233 (4)	0.5970 (4)	0.6827 (3)	0.0418 (9)
C7	0.2162 (4)	0.7193 (4)	0.6650 (3)	0.0419 (9)
C8	0.1304 (5)	0.7895 (5)	0.6067 (4)	0.0573 (12)
H8A	0.2086	0.8694	0.6053	0.086*
H8B	0.0672	0.8107	0.6502	0.086*
H8C	0.0613	0.7328	0.5278	0.086*
C9	0.8449 (4)	0.8867(4)	0.7933(4)	0.0414 (9)
C10	0.9435 (5)	1.0056 (4)	0.7786 (4)	0.0559(11)
H10	0.8951	1.0050 (1)	0.7342	0.067*
C11	1.1084 (6)	1.0620 (5)	0.8293(5)	0.0699 (14)
H11	1 1699	1 1386	0.8180	0.084*
C12	1 1844 (5)	1.0075 (5)	0.8966 (5)	0.001
H12	1 2960	1.0075 (0)	0.9315	0.087*
C13	1.0938 (5)	0.8930(5)	0.9118 (4)	0.0605 (12)
H13	1 1464	0.8563	0.9565	0.073*
C14	0.9226 (4)	0.8284(4)	0.8617(3)	0.0410 (9)
C15	0.8344 (4)	0.7091 (4)	0.8844(3)	0.0425 (9)
C16	0.9244 (6)	0.6458 (5)	0.9492 (5)	0.0648 (13)
H16A	0.8485	0.5657	0.9520	0.097*
H16B	0.9902	0.7058	1.0277	0.097*
H16C	0.9915	0.6259	0.9089	0.097*
C17	0.4664 (4)	0.8676 (4)	0.2612 (3)	0.0362 (8)
C18	0.4051 (5)	0.8508 (4)	0.1408(3)	0.0453(9)
H18	0.3634	0.9074	0.1197	0.054*
C19	0.4051 (5)	0.7535 (4)	0.0537 (4)	0.0472 (10)
H19	0.3619	0.7437	-0.0247	0.057*
C20	0.4707(5)	0.6694 (4)	0.0840(4)	0.0465 (10)
H20	0.4724	0.6042	0.0259	0.056*
C21	0.5329 (4)	0.6839 (4)	0.2007 (4)	0.0423 (9)
H21	0.5776	0.6283	0.2197	0.051*
C22	0.5311 (4)	0.7810(3)	0.2934(3)	0.0336 (8)
C23	0.5920 (4)	0.7872 (4)	0.4156 (3)	0.0378 (8)
C24	0.6642 (7)	0.6950 (5)	0.4440 (4)	0.0690 (14)
H24A	0.6945	0.7098	0.5267	0.104*
H24B	0.7576	0.7121	0.4218	0.104*
H24C	0.5861	0.6049	0.4013	0.104*
01	0.3522 (3)	0.5619 (2)	0.7790 (2)	0.0421 (6)
O2	0.3737 (4)	0.7706 (3)	0.6987 (3)	0.0619 (8)
03	0.6876 (3)	0.8377 (3)	0.7402 (3)	0.0506 (7)
04	0.6767 (4)	0.6530 (3)	0.8491 (3)	0.0633 (9)

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05	0.4601 (4)	0.9650 (3)	0.3363 (2)	0.0488 (7)
O6	0.5830 (4)	0.8684 (3)	0.5029 (3)	0.0571 (8)

Atomic displacement parameters $(Å^2)$

	U^{11}	U ²²	U^{33}	U^{12}	U^{13}	U ²³
Cul	0.0312 (3)	0.0370 (3)	0.0406 (3)	0.0157 (2)	0.0112 (2)	0.0147 (2)
Cu2	0.0444 (4)	0.0352 (3)	0.0314 (4)	0.0241 (3)	0.0137 (3)	0.0117 (3)
C1	0.038 (2)	0.039 (2)	0.039 (2)	0.0164 (16)	0.0172 (17)	0.0062 (17)
C2	0.051 (2)	0.046 (2)	0.066 (3)	0.017 (2)	0.027 (2)	0.020(2)
C3	0.049 (3)	0.062 (3)	0.087 (4)	0.007 (2)	0.034 (3)	0.021 (3)
C4	0.034 (2)	0.080 (4)	0.093 (4)	0.011 (2)	0.020 (2)	0.026 (3)
C5	0.038 (2)	0.078 (3)	0.064 (3)	0.023 (2)	0.012 (2)	0.021 (3)
C6	0.0350 (19)	0.048 (2)	0.039 (2)	0.0179 (17)	0.0128 (17)	0.0089 (18)
C7	0.0368 (18)	0.053 (2)	0.037 (2)	0.0237 (18)	0.0122 (16)	0.0116 (19)
C8	0.052 (2)	0.070 (3)	0.061 (3)	0.037 (2)	0.016 (2)	0.027 (3)
C9	0.036 (2)	0.043 (2)	0.043 (2)	0.0142 (17)	0.0169 (17)	0.0146 (19)
C10	0.052 (3)	0.050 (3)	0.066 (3)	0.017 (2)	0.023 (2)	0.027 (2)
C11	0.056 (3)	0.056 (3)	0.079 (4)	0.003 (2)	0.029 (3)	0.022 (3)
C12	0.035 (2)	0.073 (3)	0.084 (4)	0.003 (2)	0.016 (2)	0.020 (3)
C13	0.037 (2)	0.078 (3)	0.057 (3)	0.021 (2)	0.008 (2)	0.023 (3)
C14	0.0334 (19)	0.047 (2)	0.038 (2)	0.0156 (17)	0.0107 (16)	0.0122 (18)
C15	0.037 (2)	0.055 (2)	0.037 (2)	0.0239 (18)	0.0115 (17)	0.0163 (19)
C16	0.057 (3)	0.081 (3)	0.066 (3)	0.038 (3)	0.014 (2)	0.040 (3)
C17	0.0349 (18)	0.0349 (19)	0.035 (2)	0.0156 (16)	0.0097 (16)	0.0073 (17)
C18	0.048 (2)	0.050 (2)	0.038 (2)	0.0260 (19)	0.0090 (18)	0.0129 (19)
C19	0.052 (2)	0.049 (2)	0.031 (2)	0.0178 (19)	0.0122 (18)	0.0062 (19)
C20	0.054 (2)	0.039 (2)	0.041 (2)	0.0181 (18)	0.0202 (19)	0.0031 (18)
C21	0.043 (2)	0.034 (2)	0.052 (3)	0.0194 (17)	0.0206 (19)	0.0093 (18)
C22	0.0306 (17)	0.0305 (18)	0.039 (2)	0.0132 (14)	0.0133 (15)	0.0089 (16)
C23	0.0389 (19)	0.0342 (19)	0.046 (2)	0.0214 (16)	0.0170 (17)	0.0123 (17)
C24	0.100 (4)	0.081 (3)	0.055 (3)	0.071 (3)	0.024 (3)	0.023 (3)
01	0.0376 (14)	0.0390 (14)	0.0541 (17)	0.0187 (11)	0.0173 (12)	0.0189 (13)
O2	0.0529 (17)	0.065 (2)	0.074 (2)	0.0300 (15)	0.0214 (16)	0.0286 (18)
O3	0.0351 (14)	0.0563 (17)	0.065 (2)	0.0183 (13)	0.0134 (13)	0.0364 (16)
O4	0.0519 (18)	0.065 (2)	0.079 (2)	0.0271 (16)	0.0203 (16)	0.0362 (18)
05	0.079 (2)	0.0494 (16)	0.0350 (15)	0.0463 (15)	0.0201 (14)	0.0154 (13)
06	0.0668 (19)	0.0625 (19)	0.0566 (19)	0.0401 (16)	0.0235 (16)	0.0246 (16)

Geometric parameters (Å, °)

Cu1—O1	1.916 (3)	C11—C12	1.378 (7)	
Cu1—O3	1.916 (3)	C11—H11	0.9300	
Cu1—O2	1.934 (3)	C12—C13	1.375 (7)	
Cu1—04	1.947 (3)	C12—H12	0.9300	
Cu2—O5 ⁱ	1.906 (3)	C13—C14	1.423 (5)	
Cu2—O5	1.906 (3)	C13—H13	0.9300	
Cu2—O6 ⁱ	1.952 (3)	C14—C15	1.460 (6)	

Cu2—O6	1.952 (3)	C15—O4	1.311 (5)
C1—O1	1.319 (4)	C15—C16	1.518 (5)
C1—C6	1.430 (5)	C16—H16A	0.9600
C1—C2	1.432 (6)	C16—H16B	0.9600
C2—C3	1.379 (6)	C16—H16C	0.9600
С2—Н2	0.9300	C17—O5	1.325 (4)
C3—C4	1.384 (7)	C17—C18	1.417 (5)
С3—Н3	0.9300	C17—C22	1.429 (5)
C4—C5	1.378 (7)	C18—C19	1.380 (6)
C4—H4	0.9300	C18—H18	0.9300
C5—C6	1.431 (5)	C19—C20	1.401 (6)
С5—Н5	0.9300	C19—H19	0.9300
C6-C7	1 467 (6)	C_{20} C_{21}	1 380 (6)
C7-02	1.107(0)	C20—H20	0.9300
C7 - C8	1 519 (5)	$C_{20} = 1120$ $C_{21} = C_{22}$	1430(5)
C8-H8A	0.9600	C21 C22	0.9300
C8 H8B	0.9600	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1.471(5)
	0.9000	$C_{22} - C_{23}$	1.471(3) 1.213(5)
$C_0 = C_0^2$	0.9000	$C_{23} = C_{24}$	1.515(5)
$C_{3} = 0.5$	1.321(4)	C23-C24	1.520 (5)
C_{9}	1.426(5)	C_{24} H_{24} H_{24} H_{24} H_{24}	0.9000
C_{3}	1.430(0) 1.274(7)	C24—H24B	0.9000
	1.374 (7)	C24—H24C	0.9000
С10—п10	0.9300		
$01_{1}_{1}_{1}_{1}_{1}_{1}_{1}_{1}_{1}_{1$	173 38 (12)	C13_C12_H12	120.3
01 - Cu1 - 03	173.30(12)	C12 - C12 - C14	120.5
$O_1 = Cu_1 = O_2$	91.92 (12) 88.35 (12)	C12 - C13 - C14 C12 - C13 - H13	118 7
03 - Cu1 - 02	80.33(12)	C12-C13-H13	118.7
01 - Cu1 - 04	89.17(12)	C14 - C13 - H13	116.7
03 - Cu1 - 04	91.03(12) 175.72(15)	C13 - C14 - C9	117.0(4)
02-Cu1-04	1/3.72(13)	C13 - C14 - C13	119.4 (4)
03 - Cu2 - 03	180.000(1)	C9 - C14 - C13	123.0(3)
$05 - Cu2 - 06^{\circ}$	92.29 (12)	04 - C15 - C14	121.0 (3)
$05-Cu2-06^{\circ}$	87.71 (12)	04-015-016	118.1 (4)
05-Cu2-06	87.71 (12)	C14-C15-C16	120.3 (3)
05—Cu2—06	92.29 (12)	C15—C16—H16A	109.5
06 ¹ —Cu2—O6	180.000 (1)	C15—C16—H16B	109.5
01	125.1 (3)	H16A—C16—H16B	109.5
01	116.9 (4)	C15—C16—H16C	109.5
C6—C1—C2	118.0 (3)	H16A—C16—H16C	109.5
C3—C2—C1	121.9 (4)	H16B—C16—H16C	109.5
C3—C2—H2	119.1	O5—C17—C18	116.4 (3)
C1—C2—H2	119.1	O5—C17—C22	124.8 (3)
C2—C3—C4	120.5 (5)	C18—C17—C22	118.8 (3)
С2—С3—Н3	119.8	C19—C18—C17	122.2 (4)
С4—С3—Н3	119.8	C19—C18—H18	118.9
C3—C4—C5	119.5 (4)	C17—C18—H18	118.9
C3—C4—H4	120.3	C18—C19—C20	119.6 (4)
С5—С4—Н4	120.3	C18—C19—H19	120.2

C4—C5—C6	122.7 (5)	С20—С19—Н19	120.2
С4—С5—Н5	118.7	C21—C20—C19	119.7 (4)
С6—С5—Н5	118.7	С21—С20—Н20	120.2
C1—C6—C5	117.5 (4)	С19—С20—Н20	120.2
C1—C6—C7	123.1 (3)	C20—C21—C22	122.5 (4)
C5—C6—C7	119.4 (4)	C20—C21—H21	118.7
O2—C7—C6	121.4 (3)	C22—C21—H21	118.7
O2—C7—C8	118.5 (4)	C17—C22—C21	117.2 (3)
C6—C7—C8	120.1 (3)	C17—C22—C23	122.9 (3)
С7—С8—Н8А	109.5	C21—C22—C23	119.9 (3)
С7—С8—Н8В	109.5	O6—C23—C22	122.3 (3)
H8A—C8—H8B	109.5	O6—C23—C24	117.6 (4)
С7—С8—Н8С	109.5	C22—C23—C24	120.1 (3)
H8A—C8—H8C	109.5	C23—C24—H24A	109.5
H8B—C8—H8C	109.5	C23—C24—H24B	109.5
O3—C9—C14	124.7 (3)	H24A—C24—H24B	109.5
O3—C9—C10	117.0 (4)	С23—С24—Н24С	109.5
C14—C9—C10	118.3 (4)	H24A—C24—H24C	109.5
C11—C10—C9	120.8 (4)	H24B—C24—H24C	109.5
C11—C10—H10	119.6	C1—O1—Cu1	127.9 (2)
С9—С10—Н10	119.6	C7—O2—Cu1	130.4 (3)
C10—C11—C12	121.3 (4)	C9—O3—Cu1	127.4 (2)
C10-C11-H11	119.3	C15—O4—Cu1	130.0 (3)
C12—C11—H11	119.3	C17—O5—Cu2	127.9 (2)
C11—C12—C13	119.4 (4)	C23—O6—Cu2	129.0 (3)
C11—C12—H12	120.3		

Symmetry code: (i) -x+1, -y+2, -z+1.