X-ray powder diffraction data for Copper (II). bis[(2E)-3-methoxy-2-[(2,4,6-trimethylphenyl)imino]-4-[(2,4,6-trimethylphenyl)imino-κN]-3-pentanolato-κO] complex

R. Pažout,[1,a] J. Maixner,[1] A. Sokolohorskyj,[2] and J. Merna[2]
[1]*Central Laboratories, Institute of Chemical Technology Prague, Technická 5, 166 28 Prague 6, Czech Republic*
[2]*Department of Polymers, Institute of Chemical Technology Prague, Technická 5, 166 28 Prague, Czech Republic*

(Received 10 April 2014; accepted 16 July 2014)

X-ray powder diffraction data, unit-cell parameters and space group for a new bis(β-diiminato) Cu(II) complex, $C_{48}H_{62}CuN_4O_4$ are reported [a = 13.330 (3) Å, b = 11.137(2) Å, c = 8.832(2) Å, α = 78.88 (2), β = 94.73(2), γ = 114.55(2), unit-cell volume V = 1170.26 Å^3, Z = 1, and space group $P-1$]. All measured lines were indexed and are consistent with the $P-1$ space group. No detectable impurity was observed. © *2014 International Centre for Diffraction Data.*
[doi:10.1017/S088571561400075X]

Key words: unit-cell parameters, space group, X-ray powder diffraction data, bis(β-diiminato) Cu(II) complex

I. INTRODUCTION

β-diiminates are among the most important ligands in current homogeneous catalysis (Bourget-Merle *et al.*, 2002; Asay *et al.*, 2011). Diiminate complexes are prepared by the deprotonation of β-diimines followed by their coordination to metal precursor. Catalytic activity of diiminate complexes is strongly influenced by the ligand structure; therefore the preparation of novel β-diimine ligands is desirable. Copper(II). bis [(2E)-3-methoxy-2-[(2,4,6-trimethylphenyl)imi-no]-4-[(2.4.6-trimethylphenyl) imino-κN]-3-pentanolato-κO] complex (Figure 1) is a stable reaction intermediate in synthesis of keto-β-diimine ligands (Yokota *et al.*, 2002).

II. EXPERIMENTAL

A. Sample preparation

Preparation of a Cu (II) complex is based on the oxidative degradation of β-diimine by copper acetate under aerobic conditions reported by Yokota *et al.* (2002). The starting β-diimine ligand was prepared according to Feldman *et al.* (1997). Reaction with copper (II) acetate monohydrate yielded β-diiminate copper(II) acetate. which was converted to the title complex [copper(II).bis[(2E)-3-methoxy-2-[(2,4,6-trimethylphenyl)imino]-4-[(2,4,6-trimethylphenyl) imino-κN] -3-pentanolato-κO]] by treating with molecular oxygen in methanol solution for 3 days at 50 °C.

B. Diffraction data collection and reduction

The diffraction pattern for the title compound was collected at room temperature using an X'Pert PRO θ–θ powder diffractometer with parafocusing Bragg–Brentano geometry and Cu$K\alpha_1$ radiation (λ = 1.5406 Å, generator setting: 40 kV, 30 mA). An ultrafast X'Celerator detector was employed to collect X-ray diffraction data (XRD) data over the angular range from 4 to 60°2θ with a step size of 0.017° 2θ and a counting time of 20.32 s step^{-1}. The software package HighScore Plus V 3.0d of PANalytical, Almelo, Netherlands, was used to smooth the data, to fit the background, and to eliminate the $K\alpha_2$ component. The top of smoothed peak method was used to determine the peak positions and intensities of the diffraction peaks.

Automatic indexing of the experimental XRD pattern was done using DICVOL06 (Boultif and Louër, 2004).

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Automatic indexing results obtained by DICVOL06 (Table I) show that the title compound is triclinic with the space group $P-1$ and unit-cell parameters: a = 13.330 (3) Å, b = 11.137(2) Å, c = 8.832(2) Å, α = 78.88(2), β = 94.73 (2), γ = 114.55(2), unit-cell volume V = 1170.26 Å^3, and Z = 1. The figures of merits are F_{20} = 61.1(0.0080, 41) (Smith and Snyder, 1979) and M_{20} = 26.2 (de Wolf, 1968). All lines were indexed and are consistent with the $P-1$ space group.

[a]Author to whom correspondence should be addressed. Electronic mail: richard.pazout@vscht.cz

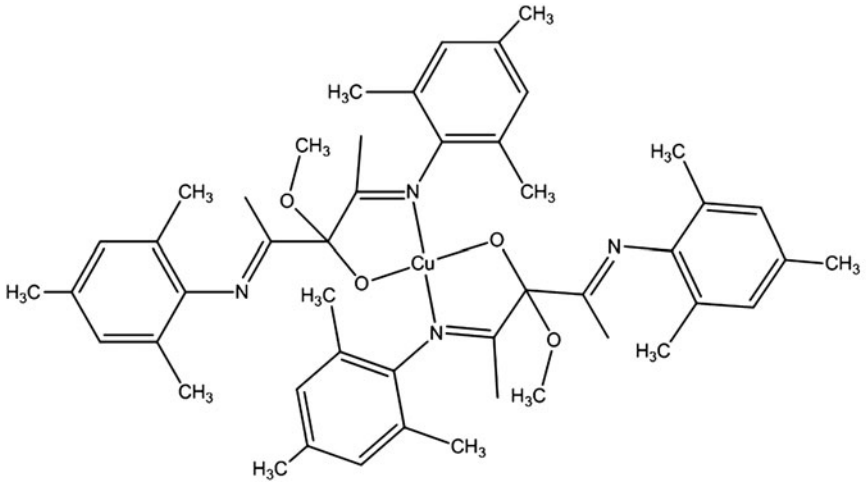

Figure 1. Structural formula of the title compound Copper (II). bis[(2E)-3-methoxy-2-[(2,4,6-trimethylphenyl)imino]-4-[(2,4,6-trimethylphenyl)imino-κN]-3-pentanolato-κO] complex.

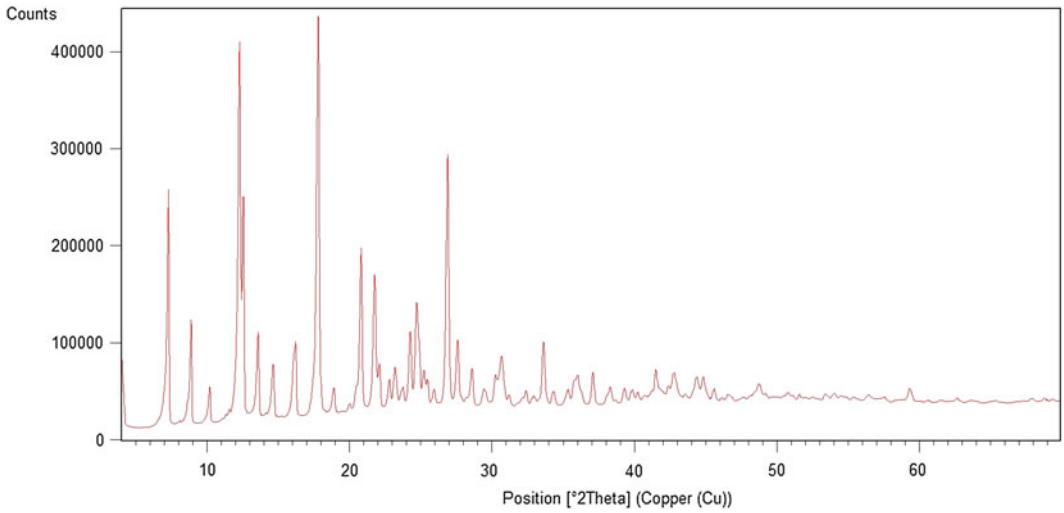

Figure 2. (Color online) X-ray powder diffraction pattern of the title compound using and Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

TABLE I. Indexed XRD data for the title compound, $C_{48}H_{62}CuN_4O_4$. Only the peaks with I_{rel} of 1 or greater are presented [$a = 13.330$ (3) Å, $b = 11.137(2)$ Å, $c = 8.832(2)$ Å, $\alpha = 78.88(2)$, $\beta = 94.73(2)$, $\gamma = 114.55(2)$, unit-cell volume $V = 1170.26$ Å^3, $Z = 1$, and space group $P-1$]. All measured lines were indexed and are consistent with the $P-1$ space group. The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

$2\theta_{obs}$ (deg)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (deg)	d_{calc} (Å)	$\Delta 2\theta$
7.284	12.127	59	1	0	0	7.283	12.128	0.001
8.845	9.989	21	0	1	0	8.857	9.976	−0.011
8.881	9.949	26	1	−1	0	8.857	9.976	0.005
10.189	8.675	9	0	0	1	10.198	8.667	−0.009
12.278	7.203	95	0	1	1	12.290	7.196	−0.012
12.554	7.046	56	1	0	−1	12.528	7.060	0.025
13.575	6.518	22	1	1	0	13.593	6.509	−0.019
14.633	6.049	14	0	1	−1	14.655	6.040	−0.021
16.102	5.500	16	1	1	1	16.067	5.512	0.035
16.203	5.466	19	1	−2	0	16.207	5.465	−0.004
17.402	5.092	8	1	−2	−1	17.421	5.086	−0.020
17.757	4.991	89	0	2	0	17.769	4.988	−0.012
17.800	4.979	100	2	−2	0	17.769	4.988	−0.009
18.898	4.692	7	0	2	1	18.896	4.693	0.002
20.006	4.435	3	2	1	0	19.989	4.438	0.017
20.489	4.331	7	0	0	2	20.479	4.333	0.010
20.815	4.264	41	0	1	2	20.855	4.256	−0.040
21.761	4.081	34	1	0	−2	21.745	4.084	0.016
22.104	4.018	12	2	−2	1	22.096	4.020	0.007

Continued

https://doi.org/10.1017/S088571561400075X Published online by Cambridge University Press

TABLE I. Continued

$2\theta_{obs}$ (deg)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (deg)	d_{calc} (Å)	$\Delta 2\theta$
22.793	3.898	8	1	2	1	22.788	3.899	0.005
23.188	3.833	11	2	1	−1	23.181	3.834	0.006
23.314	3.812	7	1	1	2	23.324	3.811	−0.010
23.600	3.767	5	1	−2	−2	23.596	3.768	0.005
23.749	3.744	6	0	1	−2	23.758	3.742	−0.009
24.265	3.665	20	3	0	−1	24.254	3.667	0.011
24.720	3.599	27	0	2	2	24.726	3.598	−0.007
24.873	3.577	17	1	−3	−1	24.863	3.578	0.010
25.217	3.529	10	2	0	−2	25.212	3.530	0.005
25.465	3.495	8	1	2	−1	25.468	3.495	−0.003
25.931	3.433	5	1	1	−2	25.936	3.433	−0.006
26.785	3.326	39	0	3	0	26.791	3.325	−0.006
26.886	3.313	63	3	−3	0	26.852	3.318	0.034
27.591	3.230	17	2	1	2	27.633	3.226	−0.042
28.150	3.167	3	2	2	1	28.159	3.166	−0.009
28.596	3.119	10	1	−3	1	28.555	3.123	0.041
29.440	3.032	5	3	1	−1	29.431	3.032	0.009
29.587	3.017	4	0	2	−2	29.558	3.020	0.030
30.252	2.952	8	3	0	2	30.252	2.952	0.000
30.668	2.913	13	0	1	3	30.652	2.914	0.016
30.773	2.903	10	1	3	1	30.785	2.902	−0.012
31.253	2.860	2	4	0	1	31.258	2.859	−0.005
31.787	2.813	1	1	0	−3	31.801	2.812	−0.014
32.121	2.784	2	1	−2	−3	32.107	2.786	0.014
32.396	2.761	4	1	1	3	32.433	2.758	−0.037
32.942	2.717	3	0	2	3	32.974	2.714	−0.032
33.621	2.664	16	3	−4	0	33.619	2.664	0.002
34.304	2.612	3	1	3	2	34.305	2.612	−0.001
35.348	2.537	4	1	1	−3	35.361	2.536	−0.013
35.804	2.506	6	2	3	1	35.829	2.504	−0.025
36.020	2.491	8	0	4	0	35.986	2.494	0.034
36.270	2.475	3	2	−4	1	36.305	2.472	−0.035
37.084	2.422	8	1	−4	1	37.053	2.424	0.031
38.051	2.363	2	3	2	2	38.073	2.362	−0.022
38.309	2.348	4	0	4	2	38.334	2.346	−0.025
38.663	2.327	1	0	2	−3	38.627	2.329	0.037
39.301	2.291	4	0	4	−1	39.274	2.292	0.028
39.848	2.260	3	5	−4	0	39.849	2.260	−0.001
40.225	2.240	3	3	1	3	40.264	2.238	−0.039
40.720	2.214	2	2	−5	−1	40.707	2.215	0.013
41.234	2.188	3	6	−2	−1	41.227	2.188	0.006
41.508	2.174	8	1	−4	−3	41.514	2.174	−0.005
42.382	2.131	4	1	0	4	42.365	2.132	0.017
42.718	2.115	7	2	−5	−2	42.710	2.115	0.008
42.830	2.110	7	1	−5	0	42.792	2.112	0.038
43.563	2.076	2	0	4	3	43.562	2.076	0.001
44.315	2.042	6	0	1	−4	44.274	2.044	0.041
44.419	2.038	6	1	−3	−4	44.462	2.036	−0.043
44.835	2.020	6	3	−5	1	44.811	2.021	0.023
45.603	1.988	3	1	1	−4	45.561	1.989	0.042
46.124	1.966	1	4	−5	1	46.135	1.966	−0.011
46.559	1.949	2	2	4	2	46.547	1.950	0.012
47.659	1.907	1	2	2	4	47.693	1.905	−0.034
48.086	1.891	2	1	4	−2	48.129	1.889	−0.043
48.722	1.867	5	2	−2	4	48.769	1.866	−0.047
49.190	1.851	2	1	5	0	49.183	1.851	0.007
49.613	1.836	1	2	−6	−1	49.632	1.835	−0.020
50.431	1.808	1	1	2	−4	50.423	1.808	0.009
50.776	1.797	2	3	−4	3	50.779	1.797	−0.003
51.159	1.784	2	2	3	−3	51.162	1.784	−0.003
51.569	1.771	2	4	−5	2	51.590	1.770	−0.020
52.511	1.741	1	6	−4	2	52.506	1.741	0.005
53.379	1.715	2	1	0	5	53.374	1.715	0.005
54.037	1.696	2	3	−6	−3	54.048	1.695	−0.012
54.482	1.683	2	1	2	5	54.485	1.683	−0.003
54.888	1.671	1	4	3	−2	54.852	1.672	0.037

Continued

https://doi.org/10.1017/S088571561400075X Published online by Cambridge University Press

TABLE I. Continued

$2\theta_{obs}$ (deg)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (deg)	d_{calc} (Å)	$\Delta 2\theta$
55.348	1.659	1	0	1	−5	55.321	1.659	0.027
56.463	1.628	2	2	−1	5	56.485	1.628	−0.022
57.502	1.601	1	1	3	5	57.501	1.601	0.001
58.361	1.580	0	7	1	1	58.327	1.581	0.033
59.336	1.556	3	0	4	5	59.320	1.557	0.016
60.584	1.527	1	7	−1	3	60.602	1.527	−0.018
62.643	1.482	1	1	−5	−5	62.663	1.481	−0.020
65.356	1.427	1	1	2	6	65.377	1.426	−0.022
67.849	1.380	1	1	6	4	67.869	1.380	−0.020
68.777	1.364	1	5	4	3	68.800	1.363	−0.022

ACKNOWLEDGEMENT

This work was supported by the Technological Agency of the Czech Republic (Project No. TA 03011394).

Asay, M., Jones, C., and Driess, M. (**2011**). "N-Heterocyclic carbene analogues with low-valent Group 13 and Group 14 elements: syntheses, structures, and reactivities of a new generation of multitalented ligands," Chem. Rev. **111**, 354–396.

Boultif, A. and Louër, D. (**2004**). "Powder pattern indexing with the dichotomy method," J. Appl. Crystallogr. **37**, 724–731.

Bourget-Merle, L., Lappert, M. F., and Severn, J. R. (**2002**). "The chemistry of beta-diketiminatometal complexes," Chem. Rev. **102**, 3031–3065.

De Wolff, P. M. (**1968**). "A simplified criterion for the reliability of a powder pattern indexing," J. Appl. Crystallogr. **1**, 108–113.

Feldman, J., McLain, S. J., Parthasarathy, A., Marshall, W. J., Calabrese, J. C., and Arthur, S. D. (**1997**). "Electrophilic metal precursors and a beta-diimine ligand for nickel(II)- and palladium(II)-catalyzed ethylene polymerizations," Organometallics **16**, 1514–1516.

Smith, G. S. and Snyder, R. L. (**1979**). "F_N: a criterion for rating powder diffraction patterns and evaluating the reliability of powder indexing," J. Appl. Crystallogr. **12**, 60–65.

Yokota, S., Tachi, Y., and Itoh, S. (**2002**). "Oxidative degradation of β-diketiminate ligand in copper(II) and zinc(II) complexes," Inorg. Chem. **41**, 1342–1344.

https://doi.org/10.1017/S088571561400075X Published online by Cambridge University Press