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The crystal structure of bis(μ_2 -5-chloro-*N*-(2-methyl-1-oxidopropylidene)-2-oxidobenzohydrazonate- $\kappa^5 N, O, O': N', O''$) pentakis(pyridine- $\kappa^1 N$)tricopper(II), $C_{47}H_{45}Cl_2N_9Cu_3O_6$

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.08 × 0.06 × 0.03 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.49 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	26,523, 4198, 0.085
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3394
$N(\text{param})_{\text{refined}}$:	306
Programs:	Bruker [1], SHELX [2, 3]

<https://doi.org/10.1515/ncrs-2020-0601>

Received November 16, 2020; accepted January 22, 2021;
published online February 12, 2021

Abstract

$C_{47}H_{45}Cl_2N_9Cu_3O_6$, monoclinic, $C2/c$ (no. 15), $a = 24.084(9)$ Å, $b = 12.152(5)$ Å, $c = 17.404(7)$ Å, $\beta = 109.984(4)^\circ$, $V = 4787(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0455$, $wR_{\text{ref}}(F^2) = 0.1363$, $T = 298$ K.

CCDC no.: 2057941

The molecular structure is shown in the Figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Source of material

The *N*-(5-chloro-acetylsalicyloyl)hydrazine) was synthesized according to reference [4]. A mixture of *N*-(5-chloro-acetylsalicyloyl)hydrazine) (0.196 g, 1 mmol) and acetate copper (0.186 g, 1 mmol) in 10 ml methanol was stirred at room temperature for 1 h. The mixture was filtered to remove impurities, and then left at room temperature. After a few days, yellow block crystals had formed. Yield (0.13 g, 43%).

Experimental details

The H atoms were geometrically placed ($C-H = 0.95-0.98$ Å) and refined as riding with $U_{\text{iso}}(H) = 1.2-1.5 U_{\text{eq}}(C)$. The N-bound H atoms were located in a difference Fourier map but were refined with a distance restraint of $N-H = 0.88 \pm 0.01$ Å, and with $U_{\text{iso}}(H)$ set to $1.2 U_{\text{eq}}(N)$ [1–3].

Comment

Multidentate ligands have been synthesized in recent years for the polynuclear metal complexes [4, 5]. The N atoms and O atoms can coordinated with the transition metals and rare

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Cu1	0.5000	0.79108 (5)	0.2500	0.03580 (18)
Cu2	0.36073 (2)	0.80720 (4)	0.37855 (2)	0.04015 (17)
Cl1	0.65323 (4)	0.92636 (9)	0.66705 (6)	0.0608 (3)
O1	0.52885 (10)	0.8181 (2)	0.36925 (14)	0.0441 (6)
O2	0.40102 (10)	0.8365 (2)	0.49188 (14)	0.0500 (6)
O3	0.32620 (10)	0.7873 (2)	0.25903 (14)	0.0449 (6)
N1	0.28051 (12)	0.8209 (2)	0.39057 (17)	0.0404 (6)
N2	0.42327 (11)	0.7972 (2)	0.26899 (16)	0.0331 (6)
N3	0.43176 (11)	0.8152 (2)	0.35292 (16)	0.0338 (6)
N4	0.5000	0.5938 (4)	0.2500	0.0482 (10)
N5	0.36530 (15)	0.5991 (3)	0.3945 (2)	0.0564 (8)
C1	0.45822 (14)	0.8552 (3)	0.52629 (19)	0.0390 (7)
C2	0.57809 (15)	0.8991 (3)	0.6137 (2)	0.0431 (8)
C3	0.53756 (17)	0.9051 (3)	0.6534 (2)	0.0487 (9)
H1	0.5495	0.9234	0.7086	0.058*
C4	0.47916 (16)	0.8836 (3)	0.6103 (2)	0.0482 (9)
H2	0.4521	0.8880	0.6376	0.058*
C5	0.48689 (13)	0.8262 (3)	0.39902 (19)	0.0346 (7)
C6	0.56057 (14)	0.8730 (3)	0.53209 (19)	0.0399 (7)
H3	0.5885	0.8703	0.5062	0.048*
C7	0.27292 (16)	0.8784(3)	0.4520 (2)	0.0497 (9)
H4	0.3052	0.9145	0.4886	0.060*
C8	0.50107 (14)	0.8504 (3)	0.48715 (19)	0.0354 (7)
C9	0.21897 (18)	0.8858 (4)	0.4627 (3)	0.0584 (10)
H5	0.2152	0.9264	0.5059	0.070*
C10	0.36667 (14)	0.7839 (3)	0.2260 (2)	0.0375 (7)
C11	0.23307 (16)	0.7723 (4)	0.3378 (2)	0.0538 (10)
H6	0.2373	0.7346	0.2936	0.065*
C12	0.34836 (15)	0.7644 (4)	0.1358 (2)	0.0497 (9)
H7	0.3831	0.7407	0.1230	0.060*
C13	0.17097 (18)	0.8331 (4)	0.4095 (3)	0.0696 (12)
H8	0.1343	0.8360	0.4163	0.084*
C14	0.4039 (2)	0.5553 (4)	0.4601 (3)	0.0705 (12)
H9	0.4325	0.6008	0.4952	0.085*
C15	0.3264 (2)	0.8737 (5)	0.0916 (3)	0.0791 (14)
H10A	0.2929	0.8990	0.1046	0.119*
H10B	0.3151	0.8629	0.0336	0.119*
H10C	0.3574	0.9274	0.1089	0.119*
C16	0.17845 (18)	0.7755 (5)	0.3456 (3)	0.0721 (13)
H11	0.1467	0.7391	0.3081	0.087*
C17	0.3023 (2)	0.6751 (5)	0.1093 (3)	0.0823 (16)
H12D	0.3179	0.6083	0.1380	0.123*
H12G	0.2917	0.6631	0.0515	0.123*
H12J	0.2679	0.6970	0.1215	0.123*
C18	0.4724 (2)	0.5355 (4)	0.2907 (3)	0.0695 (12)
H13	0.4521	0.5736	0.3192	0.083*
C19	0.3256 (2)	0.5334 (4)	0.3451 (3)	0.0697 (12)
H14	0.2993	0.5636	0.2975	0.084*
C20	0.5000	0.3652 (6)	0.2500	0.089 (2)
H15	0.5000	0.2887	0.2500	0.107*
C21	0.3205 (3)	0.4237 (5)	0.3587 (4)	0.0900 (17)
H16	0.2905	0.3815	0.3228	0.108*
C22	0.3599 (4)	0.3789 (5)	0.4253 (5)	0.106 (2)
H17	0.3581	0.3042	0.4357	0.127*
C23	0.4720 (3)	0.4230 (4)	0.2932 (4)	0.0884 (16)

Table 2: (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
H28	0.4529	0.3864	0.3239	0.106*
C24	0.4043 (3)	0.4446 (6)	0.4797 (4)	0.0967 (19)
H19	0.4323	0.4153	0.5263	0.116*

earth metals, so lots of the polynuclear metal complexes can be produced. The magnetic properties of the polynuclear metal complexes have been studied, which have good magnetic properties [6–8]. So lots of the polynuclear metal complexes were produced [9–10]. In this paper, one new Cu is described and the structure is similar to reference [8].

The title compound crystallizes in the monoclinic space group *C2/c* with one half of the complex in the asymmetric unit. The bond lengths of Cu–O are 1.978, 1.911 and 1.974 Å, the bond lengths of Cu–N are 1.987, 2.397, 1.912 and 2.020 Å, respectively. The bond lengths of Cu–N from the pyridine ligands are longer than the other Cu–N bond lengths, which are similar with the reference [8]. Each copper coordination environment in the complex exhibits a distorted pyramidal geometry. The trinuclear complex is centrosymmetric.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: Natural Science Foundation of Henan Province of China (No. 202300410010), Research Fund of Anyang Institute of Technology (Nos. YPY2019011, YPY2020025), Key discipline of environmental engineer of Anyang Institute of Technology.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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- $O,N,N,O':O'$ trinicke(II) – ethanol – acetonitrile (1/2/2), $C_{58}H_{86}Ni_3N_8O_{18}$. *Z. Kristallogr. NCS* 2018, 233, 495–497.
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