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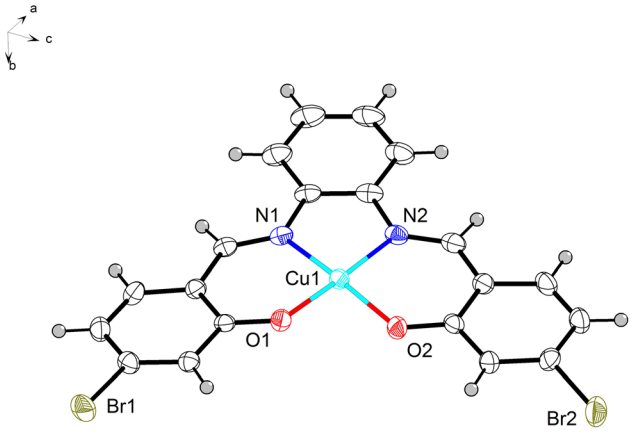
[2,2′-{Ethane-1,2-diylbis[(azanylylidene)-methanylylidene]}bis(3-bromo-2-hydroxyphenyl)]iron(III) nitrate, $C_{20}H_{12}Br_2CuN_2O_2$



https://doi.org/10.1515/ncrs-2022-0297
Received June 14, 2022; accepted August 26, 2022;
published online September 12, 2022

Abstract

$C_{20}H_{12}Br_2CuN_2O_2$, orthorhombic, *Pbca* (no. 61), $a = 7.7117(2)$ Å, $b = 18.7735(4)$ Å, $c = 24.8518(4)$ Å, $V = 3597.93(13)$ Å^3, $Z = 8$, $R_{gt}(F) = 0399$, $wR_{ref}(F^2) = 0.1125$, $T = 213$ K.

CCDC No.: 2178590

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

4-Bromo-salicylaldehyde (0.201 g, 1.0 mmol) and O-phenylenediamine (0.054 g, 0.5 mmol) were dissolved in 20 mL ethanol. The Schiff base solution was stirred at room temperature for 24 h. After this, $CuCl_2{\cdot}2H_2O$ (0.130 g, 0.75 mmol) was added and the obtained green suspension was further stirred for 2 h and filtered. The acquired green filtrate was sealed in a beaker with parafilm and kept undisturbed at room temperature. The green block crystals of the title compound were afforded after two week.

Table 1: Data collection and handling.

Crystal:	Brown plate
Size:	0.05 × 0.03 × 0.02 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	7.08 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy, ω
θ_{max}, completeness:	75.4°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11,830, 3438, 0.045
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2901
$N(param)_{refined}$:	244
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Experimental details

Data reduction was performed using the program SAINT and empirical absorption corrections were made using SADABS [1]. The structure was solved with the Olex2 program [2] as an interface together with the SHELXT and SHELXL programs [3, 4]. All H atoms were placed in geometrically idealized positions.

Comment

Schiff base is a compound containing imine or methylimine group formed via the dehydration-condensation reaction with aldehydes or carbonyl and amino compounds as reaction substrates [5–8]. The change of substituents can lead to the formation of various structures. Herein, as a part of our current research interest, we report on the exploration of the regulating effect of Schiff base ligands on transition metal complexes.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.06085 (8)	0.67436 (3)	0.33293 (2)	0.04382 (17)
Br2	0.81014 (8)	0.69047 (3)	0.73837 (2)	0.04692 (18)
Cu1	0.36991 (8)	0.47808 (3)	0.55158 (2)	0.02508 (17)
O1	0.3243 (4)	0.54688 (15)	0.49664 (11)	0.0273 (6)
O2	0.4697 (4)	0.55284 (16)	0.59301 (11)	0.0311 (6)
N1	0.2409 (4)	0.40224 (18)	0.51554 (13)	0.0259 (7)
N2	0.4143 (5)	0.40492 (19)	0.60592 (13)	0.0278 (7)
C1	0.1200 (5)	0.4771 (2)	0.44541 (15)	0.0253 (8)
C2	0.2120 (5)	0.5408 (2)	0.45785 (15)	0.0238 (8)
C3	0.1803 (5)	0.6010 (2)	0.42457 (15)	0.0268 (8)
H3	0.2326	0.6449	0.4329	0.032*
C4	0.0737 (6)	0.5956 (2)	0.38033 (16)	0.0300 (9)
C5	−0.0159 (6)	0.5336 (3)	0.36774 (16)	0.0326 (9)
H5	−0.0901	0.5313	0.3378	0.039*
C6	0.0079 (6)	0.4758 (2)	0.40061 (16)	0.0322 (9)
H6	−0.0528	0.4336	0.3930	0.039*
C7	0.1390 (6)	0.4115 (2)	0.47461 (16)	0.0280 (8)
H7	0.0726	0.3723	0.4632	0.034*
C8	0.2550 (6)	0.3359 (2)	0.54244 (17)	0.0293 (9)
C9	0.1822 (7)	0.2719 (2)	0.5246 (2)	0.0405 (11)
H9	0.1211	0.2705	0.4919	0.049*
C10	0.2003 (8)	0.2104 (3)	0.5554 (2)	0.0475 (13)
H10	0.1501	0.1675	0.5437	0.057*
C11	0.2915 (8)	0.2124 (3)	0.6029 (2)	0.0465 (13)
H11	0.3021	0.1708	0.6237	0.056*
C12	0.3681 (7)	0.2748 (3)	0.6207 (2)	0.0406 (11)
H12	0.4332	0.2751	0.6526	0.049*
C13	0.3480 (6)	0.3373 (2)	0.59075 (18)	0.0309 (9)
C14	0.4934 (6)	0.4166 (2)	0.65139 (17)	0.0314 (9)
H14	0.5062	0.3775	0.6747	0.038*
C15	0.5616 (6)	0.4822 (2)	0.66904 (15)	0.0290 (9)
C16	0.5476 (5)	0.5469 (2)	0.63900 (15)	0.0272 (8)
C17	0.6244 (6)	0.6097 (2)	0.66107 (15)	0.0314 (9)
H17	0.6167	0.6532	0.6425	0.038*
C18	0.7095 (6)	0.6061 (3)	0.70954 (16)	0.0341 (10)
C19	0.7245 (6)	0.5432 (3)	0.73969 (17)	0.0369 (11)
H19	0.7831	0.5427	0.7729	0.044*
C20	0.6511 (6)	0.4829 (3)	0.71920 (17)	0.0370 (11)
H20	0.6599	0.4403	0.7389	0.044*

Single-crystal X-ray structural analysis revealed that the title compound crystallizes in the orthorhombic crystal system (see the Abstract). The divalent Cu ion located at a general position was found to be planar four-coordinated. A further analysis indicates that the copper center occupied a N_2O_2 compartment of a salen type bromo-substituted tetradentate ligand. The dihedral angles between the three benzene rings of Schiff base ligand are 9.1°(16), 7.5°(12) and 1.7°(174) respectively, therefore the whole molecule presents planar configuration. All bond lengths are in the expected ranges [9].

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

Research funding: National Nature Science Foundation of China (No. 31760257, 21761017), the Program for Innovative Research Team (in Science and Technology) in the Universities of Yunnan Province (IRTSTYN), the Recruitment Program of Yunnan Province Experts Provincial Young Talents (2019HB098) and the Ten-Thousand Talents Program of Yunnan Province (YNWR-QNBJ-2018-273).

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

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