Scientific paper

Synthesis, Crystal Structures and Urease Inhibition of Three Copper(I) Complexes Derived from 2-(2,4-Dichlorobenzydene)hydrazine-1-carbothioamide

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Received: 09-17-2025

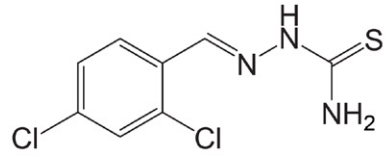
Abstract

Reaction of 2-(2,4-dichlorobenzydene)hydrazine-1-carbothioamide (L), triphenylphosphine with cuprous chloride, cuprous bromide and cuprous iodide, respectively, under the nitrogen atmosphere, afforded three new copper(I) complexes $[CuClL(Ph_3P)_2]\cdot MeCN$ (**1**), $[CuBrL(Ph_3P)_2]\cdot 0.5EtOEt$ (**2**) and $[CuIL(Ph_3P)_2]$ (**3**). The complexes have been characterized by means of single crystal X-ray crystallography and XPS. The ligand L in its neutral form coordinates to the Cu ion *via* S atom. The Cu atom in each complex is coordinated by one L, two PPh_3 and one halide ligands, to form tetrahedral coordination. The XPS results are shown that the chemical valence state of copper in the product is +1. The complexes were evaluated for their urease inhibitory activity, and gave satisfactory results (IC_{50} = 13–19 μmol L^{-1}).

Keywords: Copper(I) complexes, thiosemicarbazones, triphenylphosphine, crystal structures, urease inhibition.

1. Introduction

Thiosemicarbazones constitute a wide variety of compounds that share the $R^1R^2C{=}N{-}NH{-}C(S){-}NH_2$ framework. The compounds exist as thione-thiol tautomers and can bind to metal ions *via* a variety of coordination modes in the thione or in the anionic forms. The interest in thiosemicarbazone comes from not only bonding modes and structural diversity, but also their coordination and pharmacological activities.[1] Thiosemicarbazones have been explored as antiviral, antifungal, and antibacterial agents, etc.[2] Metal complexes of this type of ligands have shown potential biological activities.[3] Copper(II) complexes with Schiff base ligands have shown potential urease inhibitory activities.[4] However, copper(I) complexes with thiosemicarbazones have rarely studied for their urease inhibitory activity. Tertiary phosphines are known to play an important role in solubilizing Cu^I complexes of heterocyclic thioamides.[5] In this paper, three new copper(I) complexes $[CuClL(Ph_3P)_2]\cdot MeCN$ (**1**), $[CuBrL(Ph_3P)_2]\cdot 0.5EtOEt$ (**2**) and $[CuIL(Ph_3P)_2]$ (**3**), where L is 2-(2,4-dichlorobenzydene)hydrazine-1-carbothioamide (Scheme 1), are reported.



Scheme 1. The ligand L.

2. Experimental

2. 1. Materials and Methods

2,4-Dichlorobenzaldehyde, glyoxylic acid, and Ph_3P were procured from Aladdin reagent Ltd. The ligand 2-(2,4-dichlorobenzydene)hydrazine-1-carbothioamide was prepared according to the literature methods.[6] Copper(I) chloride, copper(I) bromide, and copper(I) iodide were prepared by reduction of $CuSO_4\cdot 5H_2O$ using SO_2 in the presence of NaCl, NaBr and NaI in water.[7] C, H, and N

elemental analyses were obtained with a Perkin Elmer 2400 analyzer. X-ray diffraction was carried out at a Bruker SMART 1000 CCD area diffractometer. XPS analysis was performed using a Thermo Scientific K-Alpha spectrometer; IR measurements were carried out with an FTIR-650 Fourier transform infrared spectrometer.

2. 2. Synthesis of the Complexes

2. 2. 1. [CuClL(Ph_3P)$_2$]·MeCN (1)

Under the protection of nitrogen, to CuCl (0.033 g, 0.33 mmol) suspended in acetonitrile (30 mL) was added solid PPh_3 (0.086 g, 0.30 mmol), and the contents were stirred for 24 h, followed by the addition of a solution of ligand L (0.082 g, 0.30 mmol), and continued stirring for another 4 h. The clear solution formed was filtered and allowed to evaporate at room temperature, and upon evaporation, a yellow crystalline product was formed. Yield: 0.017 g (58%). Anal. Calcd. for $C_{46}H_{40}Cl_3CuN_4P_2S$: C, 60.60, H, 4.31, N, 6.14; Found: C, 60.46, H, 4.25, N, 6.18. IR data (cm^{-1}): 3463, 3322, 3048, 1585, 1546, 1473, 1432, 1380, 1347, 1274, 1139, 1095, 871, 821, 744, 694, 516.

2. 2. 2. [CuBrL(Ph_3P)$_2$]·0.5EtOEt (2)

This complex was prepared by the similar method as complex **1**, with CuCl replaced by CuBr (0.047 g, 0.33 mmol), and with a few drops of EtOEt was diffused into the solution. Yield: 0.024 g (51%). Anal. Calcd. for $C_{90}H_{79}Br_2Cl_4Cu_2N_6O_{0.5}P_4S_2$: C, 57.83, H, 4.26, N, 4.50; Found: C, 57.66, H, 4.18, N, 4.58. IR data (cm^{-1}): 3465, 3320, 3048, 2973, 2850, 2514, 2360, 1793, 1585, 1540, 1471, 1430, 1376, 1270, 1139, 1095, 1047, 871, 819, 744, 694, 516.

2. 2. 3. [CuIL(Ph_3P)$_2$] (3)

This complex was prepared by the similar method as complex **1**, with CuCl replaced by CuI (0.063 g, 0.33 mmol), and MeCN replaced by EtOH. Yield: 0.018 g (62%). Anal. Calcd. for $C_{44}H_{37}Cl_2CuIN_3P_2S$: C, 54.87, H, 3.87, N, 4.36; Found: C, 54.66, H, 3.76, N, 4.45. IR data (cm^{-1}): 3471, 3311, 3106, 2514, 2364, 1793, 1589, 1540, 1471, 1473, 1432, 1380, 1270, 1139, 1095, 1047, 871, 819, 742, 692, 516.

2. 3. X-ray Crystallography

X-ray diffraction was carried out at a Bruker APEX II CCD area diffractometer equipped with MoKα radiation (λ = 0.71073 Å). The collected data were reduced with SAINT,[8] and multi-scan absorption correction was performed using SADABS.[9] Structures of the complexes were solved by direct method, and refined against F^2 by full-matrix least-squares method using SHELXTL.[10] All of the non-hydrogen atoms were refined anisotropically. The amino hydrogen atoms were located from electronic density maps and refined isotropically, with N–H and H···H distances restrained to 0.90(1) and 1.45(2) Å, respectively. The remaining hydrogen atoms were placed in calculated

Table 1. Crystallographic data for the complexes

	1	**2**	**3**
Empirical Formula	$C_{46}H_{40}Cl_3CuN_4P_2S$	$C_{90}H_{79}Br_2Cl_4Cu_2N_6O_{0.5}P_4S_2$	$C_{44}H_{37}Cl_2CuIN_3P_2S$
Formula weight	912.71	1869.29	963.11
T/ K	296(2)	296(2)	296(2)
Crystal system	Triclinic	Triclinic	Monoclinic
Space group	*P*$\bar{1}$	*P*$\bar{1}$	$P2_1/c$
a / Å	9.3819(17)	10.0299(14)	12.1358(12)
b / Å	13.0196(16)	13.6474(19)	24.5255(16)
c / Å	18.236(2)	16.796(2)	14.6827(12)
α / °	89.295(2)	102.989(2)	90
β / °	84.975(2)	95.829(2)	96.469(1)
γ / °	83.242(2)	99.947(2)	90
V / Å^3	2203.5(5)	2182.8(5)	4342.3(6)
Z	2	1	4
F(000)	940	957	1936
D_c / g cm^{-3}	1.376	1.428	1.473
μ(Mo *Kα*) / mm^{-1}	0.835	1.696	1.493
Measured reflections	11938	11233	21900
Unique reflections	8495	7628	7650
Observed reflections ($I \geq 2\sigma(I)$)	5102	5874	6148
Parameters	512	521	496
Restraints	15	24	4
Goodness of fit on F^2	1.018	1.051	1.087
R_1, wR_2 [$I > 2\sigma(I)$]	0.0491, 0.1049	0.0384, 0.0991	0.0331, 0.0849
R_1, wR_2 (all data)	0.0970, 0.1226	0.0555, 0.1043	0.0464, 0.0903

positions and constrained to ride on their parent atoms. The C45, C46 and O1 atoms of EtOEt solvent molecule were refined with an isotropic restraint behavior by using ISOR command. The crystallographic data and refinement parameters for the complexes are listed in Table 1.

2. 4. Measurement of *Jack Bean* Urease Inhibitory Activity

The measurement of urease activity was carried out according to the literature reported by Tanaka.[11] Generally, the assay mixture, containing 25 μL of *Jack bean* urease (10 kU L^{-1}) and 25 μL of the tested compounds of different concentrations (dissolved in DMSO/H_2O mixture (1:1 v/v)), was preincubated for 1 h at 37 °C in a 96-well assay plate. After preincubation, 200 μL of 100 mM HEPES [*N*-(2-hydroxyethyl)piperazine-*N*′- (2-ethanesulfonic acid)] buffer pH = 6.8 containing 500 mmol L^{-1} urea and 0.002% phenol red were added and incubated at 37 °C.[12] The reaction was measured by micro plate reader (570 nm), which was required to produce enough ammonium carbonate to raise the pH of a HEPES buffer from 6.8 to 7.7, the endpoint being determined by the color of phenol red indicator.[13]

3. Results and Discussion

3. 1. Synthesis

Copper(I) halid (X = Cl, Br, I) was reacted with PPh_3 under protection by nitrogen to form a copper-phosphine complex, which was then suspended in organic solvent followed by the addition of L to give the products (Scheme 2).

3. 2. Structure Description of the Complexes

Molecular structures of complexes **1**, **2** and **3** are shown as Figures 1, 2 and 3, respectively. Table 2 gives the important bond distances and angles. Complex **1** contains an acetonitrile molecule, and complex **2** contains half ethyl ether molecule. The solvent molecules are linked to the complex molecules through hydrogen bonds. Structures of the three complexes are similar, except for the halide ligand. In each complex, the Cu atom is coordinated by one S atom of L, one halide ligand (Cl for **1**, Br for **2**, I for **3**), and two P atoms from two PPh_3 ligands, to form a tetrahedral coordination. The bond angles are in the ranges of 103.93(4)–124.61(4)° (**1**), 100.80(2)–125.01(3)° (**2**), and 103.63(2)–125.99(3)° (**3**), indicating the tetrahedral geometries are distorted from ideal model. The distortion is in view of the stereochemical requirements of bulky PPh_3 ligands.[14] The Cu-S, Cu-P and Cu-X bond distances lie in the ranges 2.33–2.43, 2.26–2.29 and 2.34–2.45 Å, respectively (Table 2), which are similar to those observed in literature.[3] The S=C bond distances lie in the range 1.66–1.73 Å and are close to those (1.64–1.71 Å) observed in literature.[2] Crystals of complexes **1** and **2** are stabilized along *a* axis, with no obvious short contact. Crystal of complex **3** is stabilized by C–H···I hydrogen bonds.

Table 2. Selected bond distances (Å) and angles (°) for the complexes

	1	**2**	**3**
Cu1–P1	2.2810(10)	2.2892(9)	2.2630(8)
Cu1–P2	2.2646(10)	2.2603(8)	2.2816(8)
Cu1–X	2.3466(11)	2.5671(6)	2.6543(4)
Cu1–S1	2.3616(11)	2.3343(9)	2.3822(8)
P2–Cu1–P1	124.61(4)	125.01(3)	125.99(3)
P2–Cu1–X	103.93(4)	103.75(2)	108.37(2)
P1–Cu1–X	107.36(4)	100.80(2)	103.63(2)
P2–Cu1–S1	105.89(4)	108.65(3)	104.32(3)
P1–Cu1–S1	106.91(4)	106.24(3)	104.09(3)
X–Cu1–S1	107.09(4)	112.02(3)	109.87(2)
	X = Cl3	X = Br1	X = I1

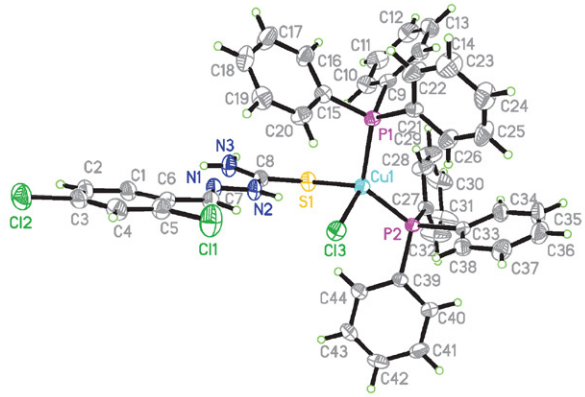
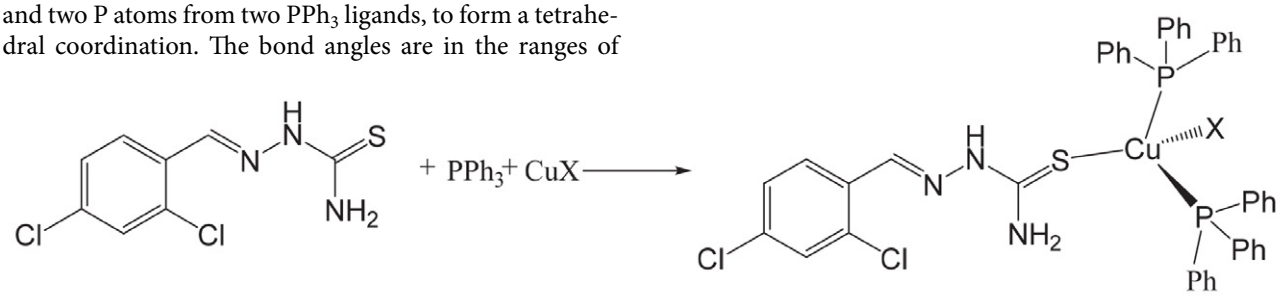


Figure 1. A perspective view of **1** with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level.



Scheme 2. The synthetic procedure for the complexes. X = Cl (**1**), Br (**2**), and I (**3**).

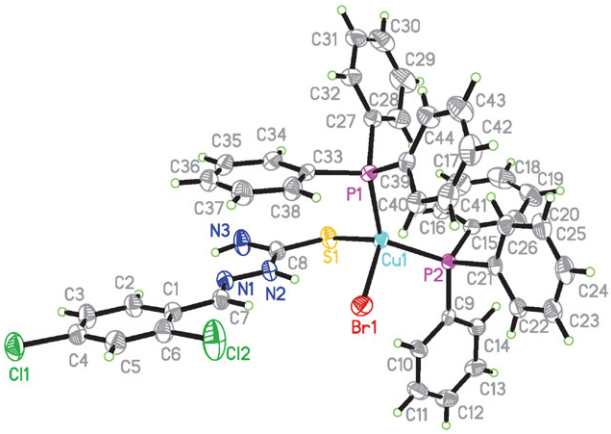


Figure 2. A perspective view of **2** with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level.

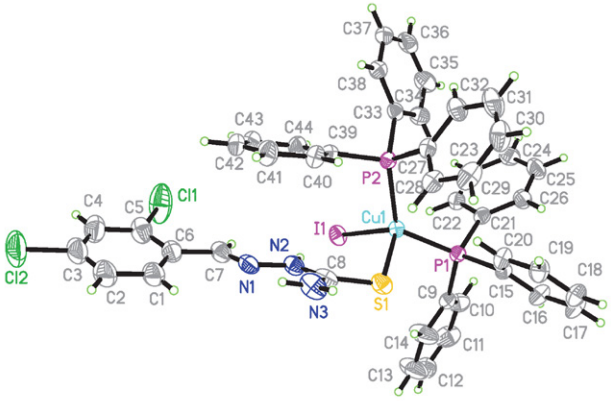


Figure 3. A perspective view of **3** with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level.

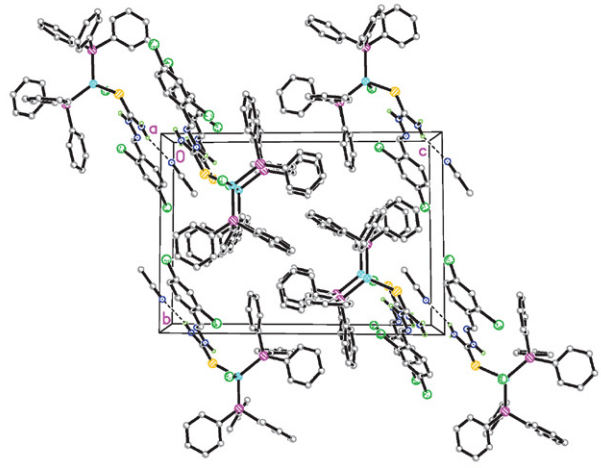


Figure 4. Molecular packing structure of **1**, viewed along *a* axis.

3. 3. X-ray Photoelectron Spectroscopy

The XPS results are shown in Figures 7–9. In the high-resolution Cu 2*p* spectrum, two intense peaks appear at 931.7 eV and 951.6 eV, corresponding to Cu $2p^{3/2}$ and

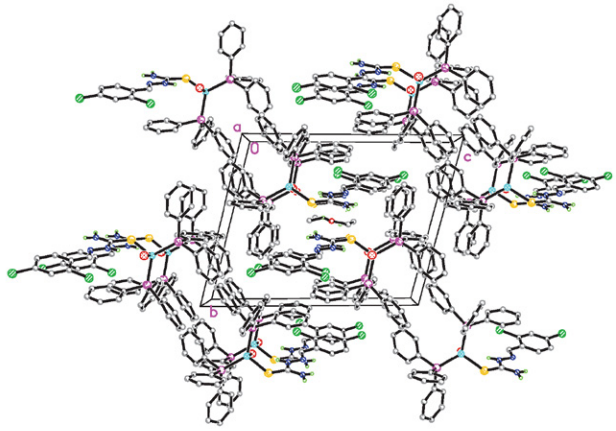


Figure 5. Molecular packing structure of **2**, viewed along *a* axis.

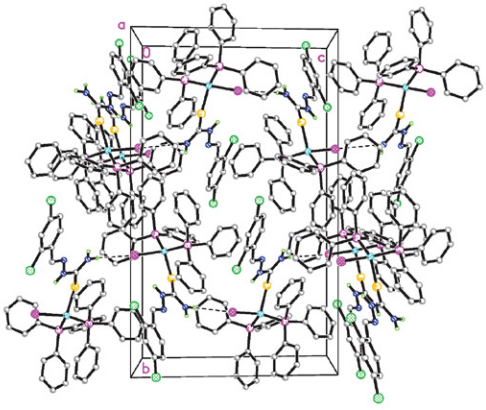


Figure 6. Molecular packing structure of **3**, viewed along *a* axis.

Cu $2p^{1/2}$ of Cu^{+}, respectively.[15] Furthermore, Cu^{2+} has a typical satellite peak at a binding energy of 942.0 eV,[16] which is not observed in the current XPS spectrum. Therefore, it can be concluded that the chemical valence state of copper in the product is +1.

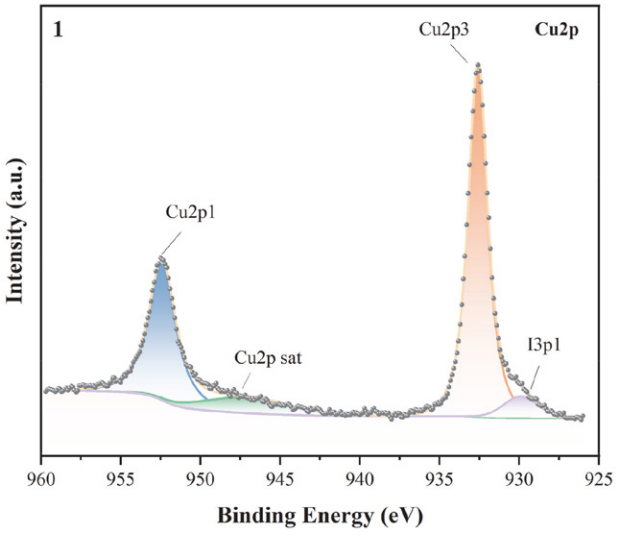


Figure 7. XPS spectrum of compound **1**

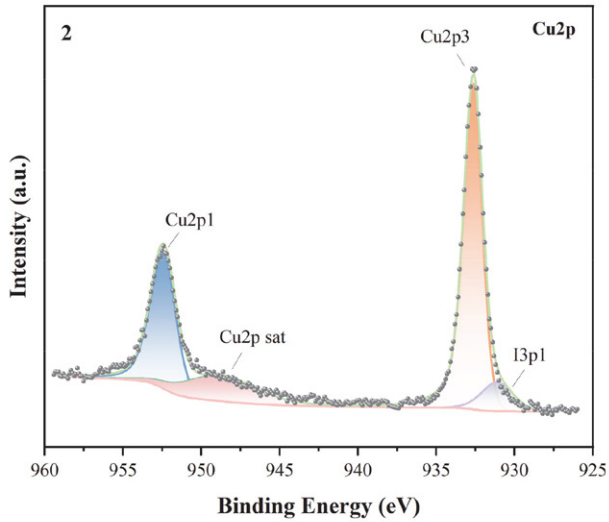


Figure 8. XPS spectrum of compound **2**

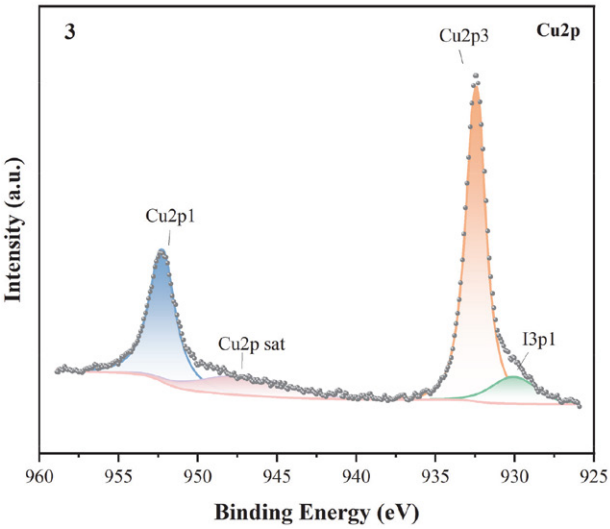


Figure 9. XPS spectrum of compound **3**

3. 4. Urease Inhibitory Activity

IC_{50} values of the activities are given in Table 3. The free ligand has no activity on urease. The three copper complexes have good inhibitory activity on Jack bean urease, with IC_{50} values of 13–19 μmol L^{-1}, which are better than acetohydroxamic acid. Interestingly, the order of the activity of the complexes is 1 > 2 > 3. Thus, the halide ligands may influence the activity, and Cl is a preferred group for the designation of urease inhibitors. The three complexes may be used as potential urease inhibitors. The urease inhibitory activity of this complex is superior to that of copper(II) complexes with various ligands: the copper complex of 2-[(2-piperidin-1-yldiacetamido)]phenol (IC_{50} = 13.5–19.9 μmol L^{-1}),[17] the Schiff base ligand *N*-(5- fluoro-2-hydroxybenzylidene)-2-amino-4-methylbenzoic acid (IC_{50} = 12.02–15.54 μmol L^{-1}),[18] the *N*-hydroxyethyl-*N*-benzimidazolylmethylacetamidediacetic acid ligand (IC_{50} = 33–37 μmol L^{-1}),[19] and the copper complex of 2-{[2-(2- hydroxyaminoethylamino)] methyl}-4-nitrophenol (IC_{50} = 24.2–24.3 μmol L^{-1}).[20] However, there is still a certain difference compared with the copper(I/II) complex of 2-((2-(dimethylamino)ethyl) methyl)-5-fluorophenol (IC_{50} = 0.4–2.2 μmol L^{-1}).[21]

Table 3. Inhibition of urease by the tested materials

The tested materials	IC_{50} (μmol L^{-1})
1	13.1 ± 0.3
2	15.2 ± 0.2
3	18.7 ± 0.5
L	–
$CuCl_2$	8.37 ± 0.62
Acetohydroxamic acid	23.7± 1.3
Ref 17	16.7 ± 3.2
Ref 18	13.78 ± 1.76
Ref 19	35 ± 2
Ref 20	24.25 ± 0.05
Ref 21	1.3 ± 0.9

4. Additional Information

CCDC 2488615 (**1**), 2488604 (**2**) and 2488605 (**3**) contain the supplementary crystallographic data. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

5. Funding

This work was financially supported by Ningbo Key Laboratory of Agricultural Germplasm Resources Mining and Environmental Regulation, College of Science and Technology, Ningbo University.

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Povzetek

Pri reakciji 2-(2,4-diklorobenziden)hidrazin-1-karbotioamida (L) in trifenilfosfina z bakrovim(I) kloridom, bakrovim(I) bromidom oziroma bakrovim(I) jodidom v dušikovi atmosferi so nastali trije novi bakrovi(I) kompleksi $[CuClL(Ph_3P)_2]\cdot MeCN$ (**1**), $[CuBrL(Ph_3P)_2]\cdot 0.5EtOEt$ (**2**) in $[CuIL(Ph_3P)_2]$ (**3**). Kompleksi so bili okarakterizirani z monokristalno rentgensko difrakcijo in XPS. Ligand L v svoji nevtralni obliki koordinira Cu ion preko S atoma. Atom Cu v vsakem kompleksu je koordiniran z enim ligandom L, dvema ligandoma PPh_3 in enim halogenidnim ligandom, kar tvori tetraedrično koordinacijo. Rezultati XPS kažejo, da je oksidacijsko število bakra v produktu +1. Kompleksi so izkazali zadovoljive rezultate pri inhibicijski aktivnosti ureaze (IC_{50} = 13–19 μmol L^{-1}).