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SPECTROSCOPIC, BIOLOGICAL ACTIVITY AND SCANNING ELECTRON MICROSCOPE STUDIES OF CD (II) WITH TETRAZOLE-THIONE AND PHOSPHINES COMPLEXES

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Abstract: Four mixed-ligand phenyl cadmium (II) complexes containing 5-benzylthion-1H-tetrazole (LH) (1) and phosphine (2-7). These complexes fall into two groups: [CdL2(κ1-dppm)],[CdL2(PPh3)2] (2,7) and [CdL2{Ph2P(CH2)nPPh2}] {where n = 1 dppm, 2 dppe, 3 dppp, 4 dppb, (CH2)n = (Cp)2Fe} (4-6). These complexes were synthesized with moderate to high yields and characterized using various techniques. Characterization data revealed that the L- ligand coordinates as a bidentate ligand through the thion sulfur atom and nitrogen in terazole cyclic in complex (1,2), and as monodentate ligand through the nitrogen terazole cyclic in complex (3-7) adopting a tetrahedral geometry around the Cd(II) center. The crystal structure of the compound was examined by using SEM. The results showed the presence of normal nanostructures.

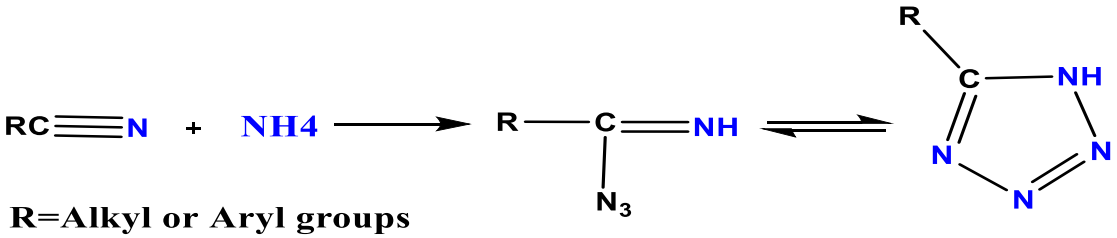
Keywords: Cd (II) complexes, biological activity, tetrazole, thiones.

1. Introduction:

Tetrazoles are heterocyclic organic compounds consisting of a pentagonal ring containing four nitrogen atoms and one carbon atom in addition to hydrogen. Their structural formula is $4N_2CH$. They are yellow solids with a distinctive odor that are soluble in water and alcohol[1]. They are widely used in the pharmaceutical field, due to their biological activity[2]. Tetrazoles exist in two tautomeric forms:



The most common method for preparing the tetrazole ring is by adding hydrazoic acid or azide to compounds containing an unsaturated nitrogen bond, such as Thiocyanate, Cyanates, Imines-Carbodi, and Cyanides. During this reaction, the intermediate compound is formed from imideazides, which undergoes a ring closure to form the tetrazole ring.[3], as below:



R=Alkyl or Aryl groups

Tetrazole-thione complexes are assigned to coordination compounds that involve the binding of tetrazole-thione ligands to a central metal atom. These ligands are derived from tetrazole compounds in which one of the nitrogen atoms is replaced by a sulfur atom (S). These complexes have attracted interest due to their diverse coordination chemistry and potential applications in various fields[4][5][6][7][8]. Tetrazole-thione ligands are characterized by the presence of a tetrazole ring (-C-N-N-N) and a thione moiety (C-S). The tetrazole ring can have substituents at different positions, influencing the coordination properties and reactivity of the ligand. Tetrazole thione ligands can coordinate with a variety of metal ions, including transition metal ions such as Cu(II), Pd(II), Pt(II), Hg(II), Zn(II), Co(II), Fe(II), etc., this coordination happens via the nitrogen atoms of the tetrazole ring and the sulfur atom of the thione group [9][10][11][12][13] . Compounds containing the tetrazole ring have occupied a wide space in the field of pharmaceutical compounds, as this ring is included in the synthesis of a number of antibiotics such as cefazolin[14] and ceftizol[15]. Tetrazole compounds have a wide spectrum of biological activity, as they are effective against diabetes [16]and effective against bacteria[17].

2. Experimental part

2.1. Materials and methods

All the chemicals, reagents and solvents for the synthesis of compounds were supplied and used without more purification. The melting point of the prepared complexes was recorded on Automatic (SMP30) melting point apparatus. The molar conductivity of 10-3 M freshly DMSO solution of prepared complexes was measured using (Starter 3100c) digital conductivity meter at 25°C. The IR spectra of the prepared complexes as KBr pellets were recorded using a Shimadzu FT-IR 8400S spectrophotometer (400-4000 cm^{-1}). NMR spectra were acquired on a Bruker 400 MHz spectrometer in DMSO-d6 as a solvent. The isolates of the pathological bacteria that were used in the research obtained from the laboratories of the Department of Life Sciences in the College of Education for Pure Sciences at the University of Tikrit.

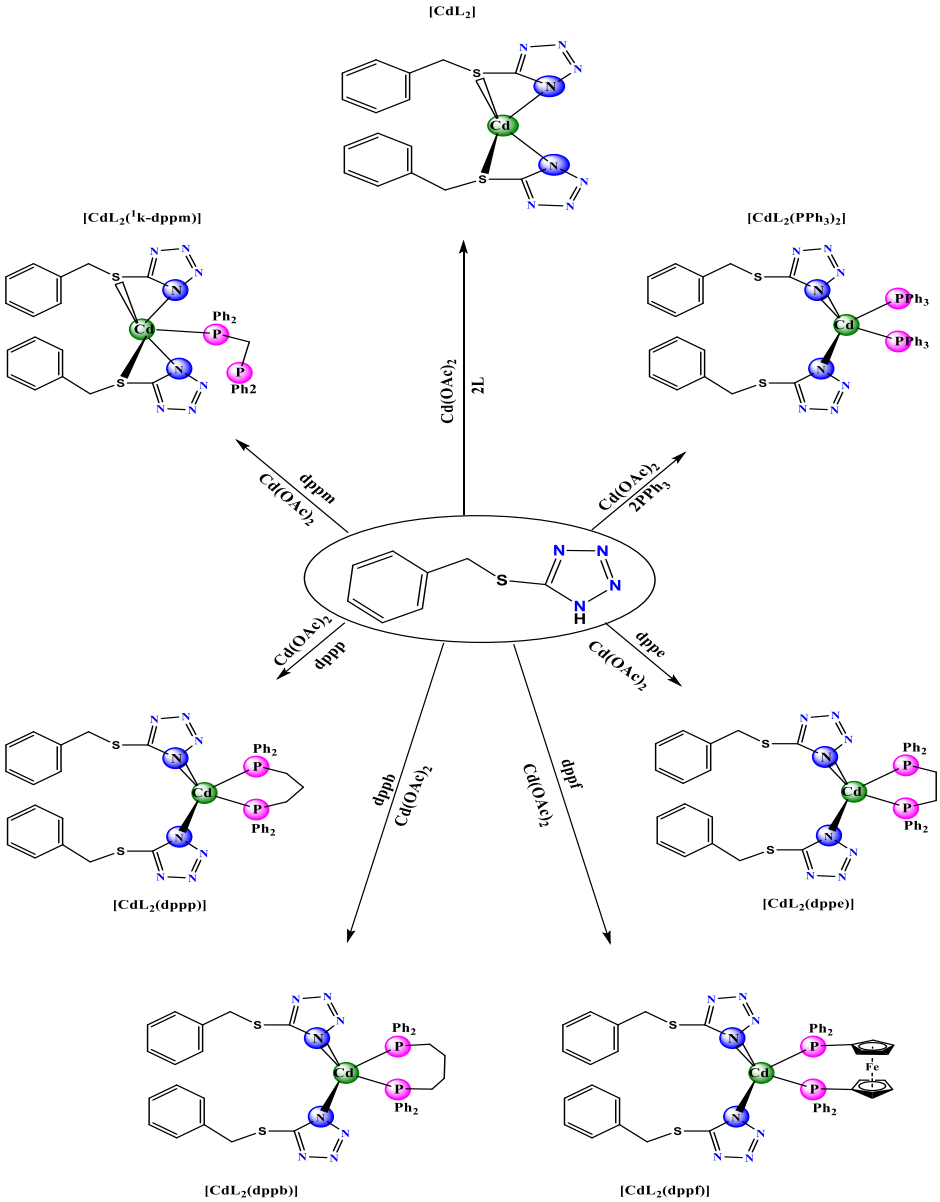
1. Preperation of [CdL_2]

A hot solution of 5-benzylthion-1H-tetrazole (LH) ligand (0.16 g; 0.86 mmol) in EtOH (5 ml) was added to a hot solution of cadmium acetate (0.100 g; 0.43 mmol) in EtOH (5 ml), with reflux. A white ppt. was formed direct. The mixture was refluxed for 3 h, then filtered off, washed with distal water and ethanol for several times, and dried under vacuum to give a white powder {Yield = 0.13 g, 61%, m.p (°C) = 240-242}.

2. Preperation of [$CdL_2(\kappa^1$-dppm)]

A hot solution of 5-benzylthion-1H-tetrazole (LH) ligand (0.16 g; 0.86 mmol) in EtOH (5 ml) was added to a hot solution of cadmium acetate (0.100 g; 0.43 mmol) in EtOH (5 ml), with reflux. A white ppt. was formed direct. The mixture was refluxed for 3 h, 1,2-bis(diphenylphosphino)methane (dppm) ligand (0.165 g; 0.43 mmol) in $CHCl_3$ (5 mL) was added to a hot suspension complex (1). The mixture was refluxed for 3 h. A clear solution was formed. The mixture was refluxed for 3 h, and left aside to slow evaporation, and then the white ppt. produced was filtered off, and dried under vacuum. The white product was recrystallized from DMSO to give a white powder {Yield = 0.118 g, 66%, m.p (°C) = 170-172}. The [CdL_2(dppe)] (3), [CdL_2(dppp)] (4), [CdL_2(dppb)] (5), [CdL_2(dppf)] (6) [$CdL_2(PPh_3)_2$] (7) was prepared and isolated by similar method starting with two moles of triphenylphosphine (Ph_3P).

Seq	Complexes	Color	m.p(°C)	Yield %		Conductivity ohm^{-1}.cm^{-2}.mol^{-1}
1	LH	white	150	-	-	5.6
2	[CdL_2]	white	240-242	0.13g	61%	14.3
3	[CdL_2(^{1}κ-dppm)]	white	170-172	0.118g	39%	18.1
4	[CdL_2(dppe)]	white	142-144	0.2g	66%	12.8
5	[CdL_2(dppp)]	white	253-255	0.15g	50%	8.3
6	[CdL_2(dppb)]	white	149-151	0.3g	76%	17.6
7	[CdL_2(dppf)]	white	164-167	0.4g	88%	16.4
8	[$CdL_2(PPh_3)_2$]	white	175-178	0.4g	86%	9.7



3. RESULTS AND DISCUSSION

3.1 Synthesis and characterization

Treatment of two molar of 5-benzylthion-1H-tetrazole ligand (L) and cadmium acetate afforded a white ppt characterized as a [CdL_2] in high yield (61%) (Scheme 1). The [CdL_2] complex was treated with equivalent mole of diphosphine {$Ph_2P(CH_2)_nPPh_2$} or two mole of Ph_3P {where n = 1 dppm, 2 dppe, 3 dppp, 4 dppb, $(CH_2)_n$ = $(Cp)_2Fe$} to afford complexes in (Scheme 2). The resulting compounds were characterized by IR, ^{1}H-NMR, ^{31}P-{^{1}H} NMR, molar conductivity measurements, and elemental analysis. The conductivity measurements data displayed that complexes have a low conductivity value and fell within the range of non-electrolyte[19] Table. 3.1.1. NMR data The ^{31}P-{^{1} H} NMR spectra of complexes (3-7) (Figure 1; Figures S1-S5) displayed a single signal, confirming equivalent phosphorus atoms and the formation of a single isomer for each complex, but [$CdL_2(\kappa^1$-dppm)] complex(2) display doublet of doublets signals meaning that two phosphorus are not equivalent, one phosphor coordinate with cadmium but the other is not[18] (Table 2).

[CdL_2]: White powder, (0.13 g), yields 61%, m.p : 240-242ºC. FTIR(KBr): 3085m ν(C-H)$_{Ar.}$, 2974m ν(C-H)$_{alip.}$, 1623s ν(C=N), 1550 υ(C=C), 1350m ν(N=N), 1240s ν(N-N), 759m ν(C-S). ^{1}H NMR: (DMSO-d6) δ(ppm): 7,46 (d, $^3J_{H-H}$ = 8.12 Hz, 4H, Ha), 7.32 (t, ($^4J_{H-H}$= 7.76 Hz,$^3J_{H-H}$= 1.26 Hz) 4H, H_b), 7.12 (t, $^4J_{H-H}$= 7.50 Hz,$^3J_{H-H}$= 1.25 Hz,2H, H_c), 4.25(s, 4H,CH_2). ^{13}C-{^{1}H}-NMR**:** 157.85 (C1), 138.76 (C2), 129.28 (s,C3), 128.76 (s,C4), 127.46 (m,C5), 36.62 (m,C6).

[$CdL_2(PPh_3)_2$]: White powder, (0.4g), yields 86%, m.p: 175-178 ºC. FTIR(KBr): 3055m ν(C-H)Ar., 2925m ν(C-H)aliph., 1633s ν(C=N), 1546s ν(C=C), 1436s ν(P-Ph), 1348s ν(N-N), 769s ν(C-S), 692m ν(P-C) .^{1}H NMR (DMSO-d6) δ(ppm): 7,49 (d, 3*J* = 8.51 Hz, 4H, Ha), 7.42 (m, 12H, H_{Ph}), 7.30 (b, 18H, H_{Ph}), 7.18 (t, $^4J_{H-H}$ = 7.69 Hz, $^3J_{H-H}$ = 1.32 Hz, 4H, Hb), 7.10 (t, $^4J_{H-H}$= 7.52 Hz,$^3J_{H-H}$= 1.24 Hz, 2H,Hc), 4.03 (s, 4H, CH_2).^{31}P{^{1}H}NMR δP=22.99ppm (s).

[$CdL_2(\kappa^1$-dppm)]: White powder, (0.118g), yields 61%, m.p : 170-172ºC,FTIR(KBr): 3066m ν(C-H)Ar., 2983m ν(C-H)aliph, 1633s ν(C=N), 1573s υ(C=C), 1429s ν(P-Ph), 1350s ν(N-N), 763m ν(C-S), 690s ν(P-C).^{1}H NMR (DMSO-d6) δ(ppm): 7,41 (d, $^3J_{H-H}$ = 7.75 Hz, 4H, Ha), 7.38 (b, 12H, H_{Ph}), 7.30 (t, $^4J_{H-H}$= 7.58 Hz,$^3J_{H-H}$= 1.27 Hz ,4H, H_b), 7.26 (t, $^4J_{H-H}$= 7.55 Hz,$^3J_{H-H}$= 1.25 Hz ,2H, H_c), 7.22 (b, 8H, H_{Ph}), 4.32 (s, 4H, CH_{2L}), 2.09 ($^4J_{P-H}$= 1.76 Hz, t, 2H, CH_{2dppm}). ^{31}P{^{1}H}NMR δP_A=27.53ppm, δP_X=-28.16ppm (dd, $^2J_{P-P}$= 50.92Hz).

[CdL_2(dppe)]: White powder, (0.2g), yields 66%, m.p: 142-144 ºC. FTIR(KBr): 3062m ν(C-H)Ar., 2927w ν(C-H)aliph., 1625s ν(C=N), 1546s υ(C=C), 1433s ν(P-Ph), 1352s ν(N-N), 729s ν(C-S), 698s ν(P-C).^{1}H NMR (DMSO-d6) δ(ppm): 7,85 (d, $^3J_{H-H}$ = 8.12 Hz, 4H, Ha), 7.70 (m, 12H, H_{Ph}), 7.54 (t, $^4J_{H-H}$= 7.49 Hz,$^3J_{H-H}$= 1.21 Hz ,4H, H_b), 7.34 (t, 2H, H_c), 7.20 (b, 8H, H_{Ph}), 4.34 (s, 4H,CH_2), 2.08 (t, 4H, CH_{2dppe}).^{31}P{^{1}H}NMR δP=29.89ppm (s). SEM: (68nm)

[CdL_2(dppp)]: White powder, (0.15g), yields 50%, m.p: 253-255 ºC. FTIR(KBr): 3056s ν(C-H)Ar., 2929w ν(C-H)aliph., 1618s ν(C=N), 1542s υ(C=C), 1429s ν(P-Ph), 1352s ν(N-N), 742m ν(C-S), 700s ν(P-C).^{1}H NMR (DMSO-d6) δ(ppm): 7,67 (d, $^3J_{H-H}$ = 7.81Hz, 4H, Ha), 7.53 (t, $^4J_{H-H}$= 7.56 Hz,$^3J_{H-H}$= 1.28 Hz ,4H, H_b), 7.39 (t, $^4J_{H-H}$= 7.51, $^3J_{H-H}$= 1.22 Hz ,2H, Hc), 7.17 (b, 20H, H_{Ph}), 4.23 (s, 4H,CH_2), 1.70 (t, 2H, CH_{2dppp}), 1.55 (t, 4H, CH_{2dppp}).^{31}P{^{1}H}NMR δP= 39.62 ppm(s).

[CdL_2(dppb)]: White powder, (0.3g), yields 76%, m.p: 149-151 ºC. FTIR(KBr): 3082m ν(C-H)Ar., 2906w ν(C-H)aliph., 1631s ν(C=N), 1550s υ(C=C), 1429s ν(P-Ph), 1348s ν(N-N), 740m ν(C-S), 694s ν(P-C).^{1}H NMR (DMSO-d6) δ(ppm): 7,66 (d, $^3J_{H-H}$ = 8.08 Hz, 4H, Ha), 7.51 (b, 12H, H_{Ph}), 7.35 (t, $^4J_{H-H}$= 7.42 Hz,$^3J_{H-H}$= 1.16Hz ,4H, H_b), 7.23 (t, $^4J_{H-H}$= 7.48,$^3J_{H-H}$= 1.18Hz, 2H, H_c), 7.06 (m, 8H, H_{Ph}), 4.48 (s, 4H,CH_{2L}), 1.33 (t, 4H, CH_{2dppb}), 1.30 (t, 4H, CH_{2dppb}).^{31}P{^{1}H}NMR δP=24.18ppm (s).

[CdL_2(dppf)]: Orang powder, (0.4g), yields 88%, m.p: 164-167 ^oC. FTIR(KBr): 3049m ν(C-H)Ar., 2939m ν(C-H)aliph., 1637s ν(C=N), 1556s υ(C=C), 1433s ν(P-Ph), 1334s ν(N-N), 752m ν(C-S), 694s ν(P-C).^{1}H NMR (DMSO-d6) δ(ppm): 7,51 (d, $^3J_{H-H}$ = 7.98 Hz, 4H, Ha), 7.42 (m, 12H, H_{Ph}), 7.33 (t, $^4J_{H-H}$= 7.40 Hz,$^3J_{H-H}$= 1.16 Hz ,4H, H_b), 7.23 (t, $^4J_{H-H}$= 7.48 Hz,$^3J_{H-H}$= 1.20Hz ,2H, H_c), 7.16 (m, 8H, H_{Ph}), 4.41 (s, 4H,CH_{2L}), 3.85(m, 4H, CH_{cp}), 3.70 (m, 4H, CH_{cp}).^{31}P{^{1}H}NMR δP= 23.12ppm (s).

IR data

The infrared spectrum of the complex [$Cd(LH)_2$] showed the disappearance of the N-H band that was present in the used ligand shown in Figure (3-3). The spectrum of the complex showed the presence of a band at the frequency (1623 cm^{-1}) dating back to the stretch of ν(C). =N)[19], the appearance of a band at (1550cm^{-1}) is attributed to the stretching of the group ν(C=C)[20], and the band at frequency (1350cm^{-1}) is due to the stretching of the group ν(N=N) [14], and the appearance of a band Also at (1240cm^{-1}) it was attributed to the ν(N-N) stretch[21], and it also showed a band at the frequency (759cm-1) that was attributed to the ν(C-S) stretch, in addition to the frequency (3085cm-1) which belongs to the ν(C-H) aromatic and the frequency (2974cm^{-1}) belongs to the aliphatic ν(C-H) stretch and the appearance of a band at frequency (426cm^{-1}) belongs to the ν(Cd-N) group [21]

While the infrared spectrum of the prepared complexes showed a new signal between the range (1433 s - 1436 s) and (m507 - m511) attributed to υ(P-Ph) and υ(P-C), respectively, and a signal within the range (1633 s (1636 s), attributed to υ(C=N) group, and a signal within the range (2925 m (2927 w), (3055 s - 3062 m) attributed to Alph.υ(C-H) and Ar υ(C-H) .

The Results of SEM and EDX

The recent studies on the geometry of coordinate compounds have shown that most of them can be systematically produced nano-sized engineering structures[22]. In this paper, the morphological appearance of the microstructure of some complexes prepared by SEM technology was studied. The SEM image of [$CdL_2(PPh_3)_2$] It showed formation of irregular sheets with a nano size of 150-225 nm (as in Fig. 3a). The EDX result showed remarkable peaks representing the atoms found in the complex (Fig. 3b).

The SEM image of [CdL_2(dppe)] showed the formation of irregular cubes, some of them were fused with a nano size of 80 −122 nm (as in Fig. 4a), and the EDX result showed remarkable peaks representing the atoms found in the complex (Fig. 4b).

While the SEM image of [CdL_2(dppp)] showed the formation of irregular clusters with a nano size of 66-137 nm (as in Fig. 5a), and the EDX result showed remarkable peaks representing the atoms found in the complex (Fig. 5b).

These results are good results in the field of coordination chemistry, as they open up prospects for the usability of complexes in a wide range of applications. Regarding the results obtained, the measurements proved the existence of regular nanostructures with high porosity, which means that these compounds are able to be used in applications requiring a high porosity surface area, such as those required in hydrogen storage applications[23]. Although the compositions of nanocomplexes were addressed infrequently the SEM results in this study are consistent with the previously published results indication the possibility to synthesize complexes containing nanostructures. It was also revealed that the particle sizes in this study were somewhat larger than those previously reported.

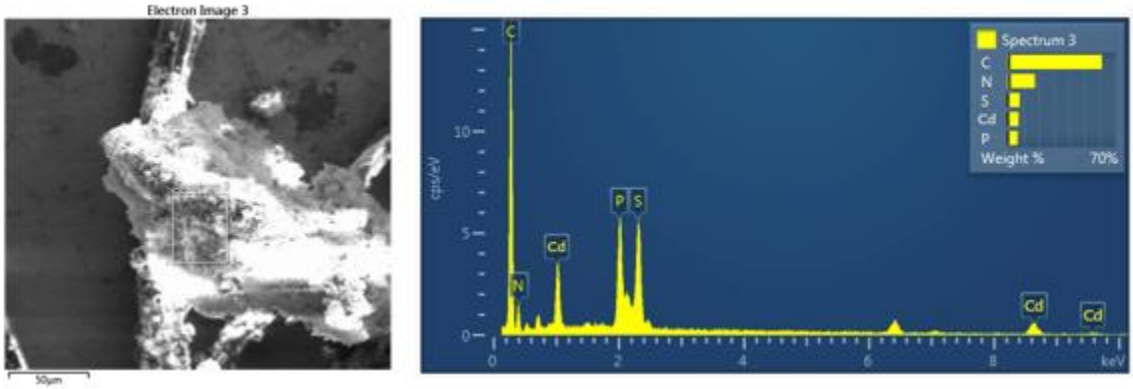
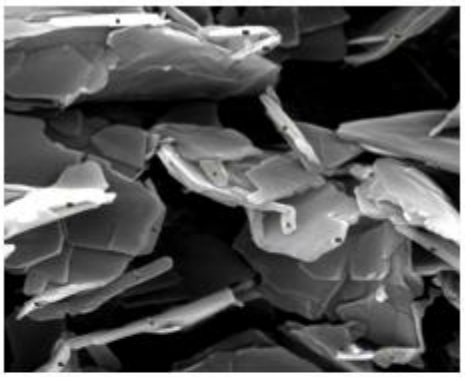


Figure 3a: The SEM measurements of $[CdL_2(PPh_3)_2]$ / 3b: The EDX measurements of $[CdL_2(PPh_3)_2]$



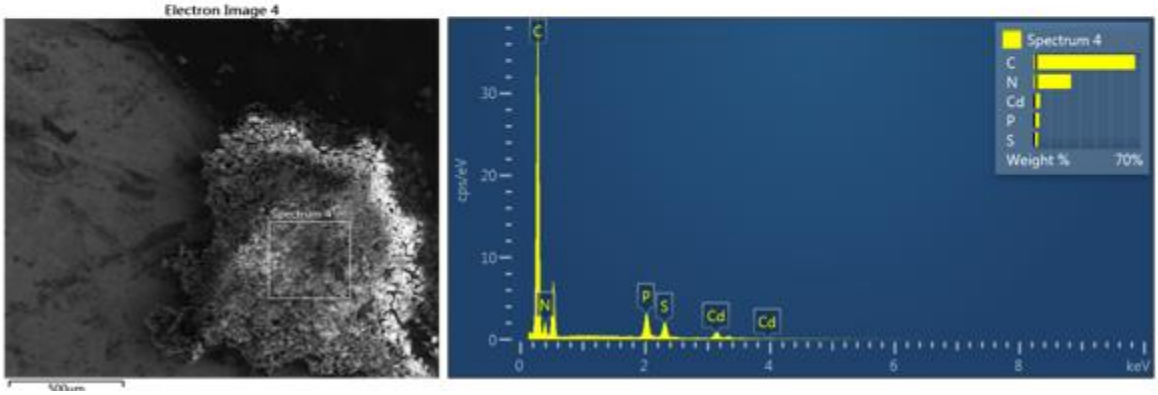


Figure 4a: The SEM measurements of $[CdL_2(dppe)]$ / 4b: The EDX measurements of $[CdL_2(dppe)]$

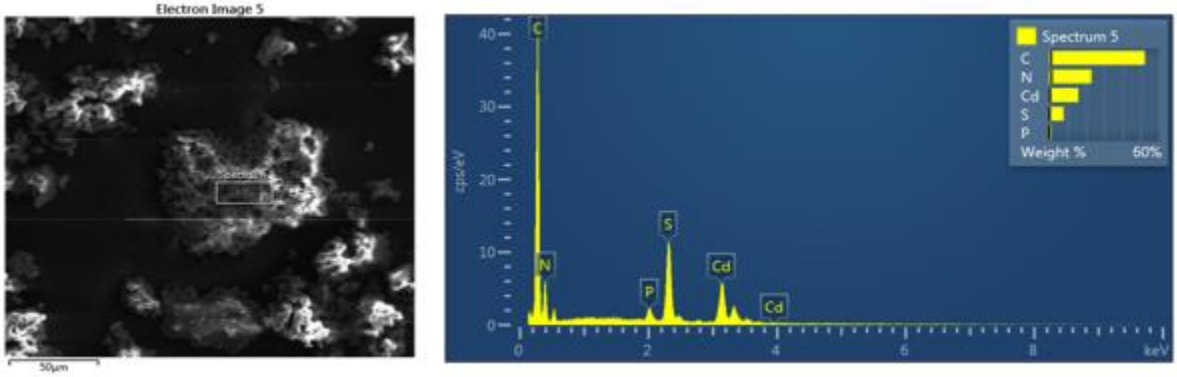


Figure 5a: The SEM measurements of $[CdL_2(dppp)]$ / 5b: The EDX measurements of $[CdL_2(dppp)]$

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