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RESEARCH ARTICLE

Synthesis and Crystal Structure Studies of Bis-[1-(*p*-methoxybenzyl)-2-(*p*-methoxyphenyl)-1*H*-benzimidazole]tetrabromidocadmate(II)

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Abstract: The complex $Cd(L)_2Br_4$, contains a dibromocadmate(II) coordinated to two benzimidazole moiety. In the crystal structure the molecules are further interconnected with weak intermolecular C-H···O hydrogen bonds generating a three-dimensional network. Further the supramolecular assembly is stabilized by Intermolecular C-H···π and π···π interactions provide additional stability to the structure. Additionally the metal ion Cd(II) exhibits a distorted tetrahedral coordination of the bromine atoms.

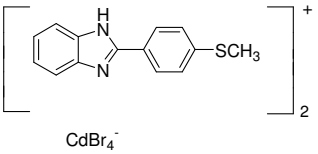
Keywords: Benzimidazole derivative, Metal ligand complex, Crystal structure, C-H…O, Weak interactions

Introduction

Benzimidazole derivatives exhibit a number of important structures in pharmaceutical chemistry because of their biological activities. They exhibit antitumor, anthelmintic, antibacterial, and fungucidal properties. In addition, benzimidazole derivatives are effective against the human cytomegalo virus (HCMV)[1] and are also efficient selective neuropeptide Y Y1 receptor antagonists[2]. As a part of our ongoing investigations of benzimidazole derivative, the benzimidazole derivative was synthesized as a ligand for complexation with cadmium metal to give the above metal complex and its crystal structure is reported.

Experimental

***Cd(L)$_2$Br$_4$*:** An ethanolic solution (15 mL) of the 2-(4-methyl sulfanyl phenyl)-1*H*-benzimidazole) (0.960 mg, 2 mmol) was added to a solution of cadmium(II) bromide (0.272 mg, 1 mmol) in ethanol (25 mL). The mixture was then treated with 48% HBr (2-3 mL) followed by liquid bromine (2-3 mL). The mixture was refluxed for nearly 6 h during which yellow crystals suitable for x-ray analysis were obtained. The crystals were collected, washed with cold ethanol and dried *in vacuum* over P_2O_5 (yield 1.23 mg, 85%).



Scheme 1. Cd(L)$_2$Br$_4$. [L=2-(4-methyl sulfanyl phenyl)-1*H*- benzimidazole]

X-Ray crystallographic analysis

The crystal structures of the complex Cd(L)$_2$Br$_4$. was determined by single crystal x-ray diffraction technique. Single crystals were obtained from the mother liquor on slow evaporation of the solvent over a week. X-ray diffraction data were collected on an oxford xcalibur diffractometer[3] equipped with an Eos CCD detector (Mo Kα radiation, graphite monochromator, λ = 0.71073 Å). Intensity data were collected using a single crystal with dimensions 0.2x0.18x0.18 mm up to a maximum of 26.81° in the ω–ϕ scan mode. The data were reduced using SAINTPLUS[4]. The structures were solved by direct methods using SHELXS-97[5] and refined with full matrix least-squares technique by using SHELXL-97[5]. The non-hydrogen atoms were refined anisotropically and all the hydrogen atoms were fixed geometrically and refined isotropically. The H atoms were placed at calculated positions in the riding model approximation (C---H 0.93Å), with their temperature factors set to 1.2 times those of the equivalent isotropic temperature factors of the parent atoms. All other non-H atoms were refined anisotropically. The R factor for observed data finally converged to R=0.029. The maximum and minimum values of residual electron density were -0.272, and 0.422eÅ^{-3}. Geometry calculations were performed using PLATON[6], ORTEP representations were prepared using ORTEP-3[7] and packing diagrams were made using CAMERON[8] incorporated in the WinGX[7] software packing. The mean plane calculation was done using the program PARST[9].

Results and Discussion

Figure 1 shows the ORTEP diagram of the molecule with thermal ellipsoids drawn at 50% probability. Figure 2 shows the hydrogen bond interactions and crystal packing in the compound. The details of crystal data and refinements are given in Table 1, 2 shows the respective hydrogen bond interactions and Table 3 shows intramolecular and intermolecular interactions for the compound.

Structure description of the complex Cd(L)$_2$Br$_4$

The title compound crystallizes in triclinic system, space group P-1 with Z=2. In the crystal structure, the metal ion, Cd(II) exhibits a distorted tetrahedral coordination of the bromine atoms and is located on a general position. Figure 1 shows the molecular structure of bis[1-(*p*-methoxybenzyl)-2-(*p*-methoxyphenyl)-1*H*-benzimidazole]tetrabromidocadmate(II){[(L)$_2$ (CdBr$_4$)]}. The N-C and N=C distances were found to be 1.339 Å and 1.355 Å respectively.

The tetrahedral bromide salt links the adjacent ligand molecules through the hydrogen bonds with bromides as acceptors Table 3. The molecules are further interconnected with weak intermolecular C–H···O hydrogen bonds generating a three-dimensional network as shown in Figure 2. Intermolecular C–H···π and π···π interactions provide additional stability to the structure Table 3. The bond lengths and bond angles for the benzimidazole moiety of the molecule are in good agreement, within the experimental errors, with those observed in other benzimidazole derivatives[10].

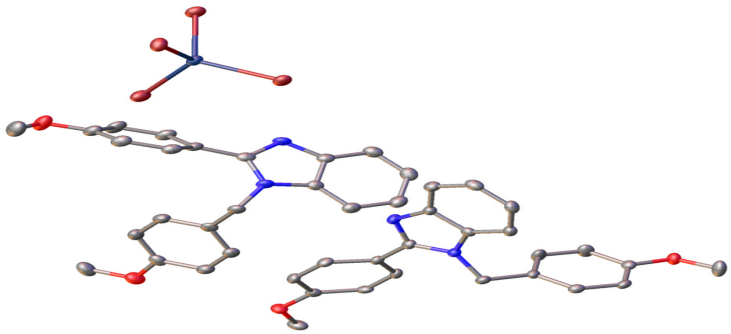


Figure 1. ORTEP view of $Cd(L)_2Br_4$ compound showing 50% probability ellipsoids

Table 1. Crystallographic data and structure refinements summary for the compound $Cd(L)_2Br_4$

CCDC number	827003
Empirical formula	C_{44} H_{40} N_4 O_4Br_4Cd
Formula weight	1120.0
Temperature/K	110(1)
Wavelength/Å	0.71073
Crystal system	Triclinic
Space group	*P*-1
Unit cell dimensions	*a* = 10.7306(2) Å *b* = 12.0680(3)Å *c* = 17.3717(4) Å α = 88.535(2)° β = 74.186(2)° *β* = 76.054(2)°
Volume (Å^3), Z	2098.69(18), 2
Density (g/mL) Calcd.	1.58
μ (1/mm)	2.947
Independent reflns	7382
No. of parameters	530
R obs, wR_2_obs	0.029, 0.075
$\Delta\rho_{min}$, $\Delta\rho_{max}$ (eÅ^{-3})	-0.272, 0.422
GooF	1.066
Measurement	Bruker SMART CCD diffractometer
Program system	SAINTPLUS
Structure determination	Direct methods (SHELXL97, SHELXS97)
Molecular graphics	ORTEP-3 (Farrugia, 1997)

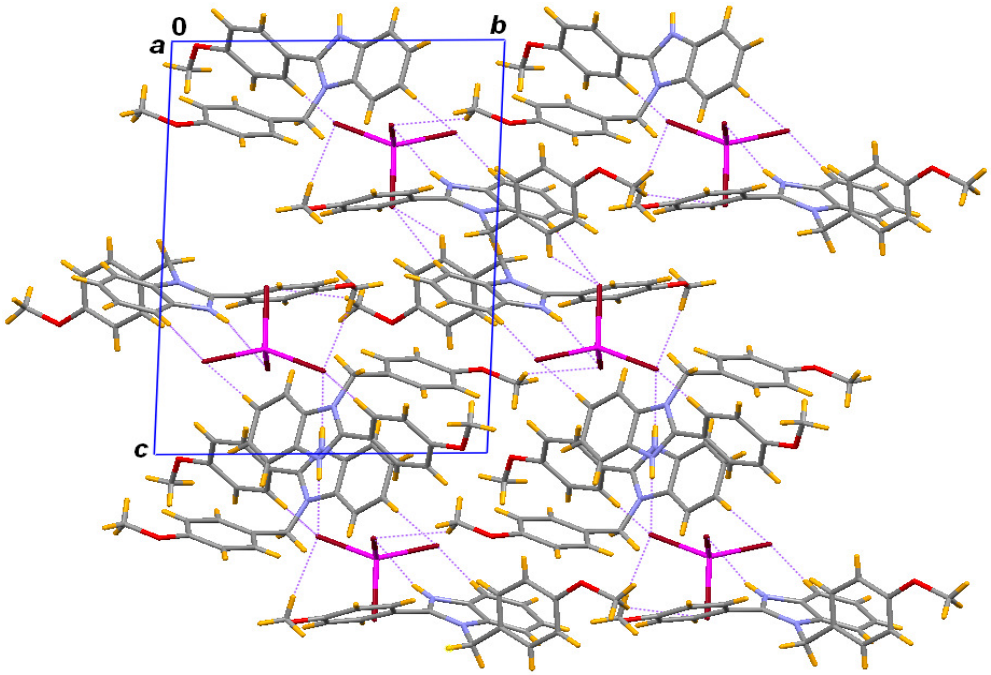


Figure 2. Crystal structure of $Cd(L)_2Br_4$, viewed along '*a*' axis. Dotted lines indicate intermolecular C─H···O interactions

Table 2. Selected bond distances and bond angles for $Cd(L)_2Br_4$

Bond distance (Å)		Bond angle (deg.)	
Cd(1)-Br(1)	2.5073 (6)	Br(1)-Cd(1)- Br(2)	112.26 (2)
Cd(1)-Br(2)	2.5409 (5)	Br(1)-Cd(1)- Br(3)	106.55 (2)
Cd(1)-Br(3)	2.5144 (6)	Br(1)-Cd(1)- Br(4)	113.09 (2)
Cd(1)-Br(4)	2.5021 (5)	Br(2)-Cd(1)-Br(3)	116.70 (2)
		Br(2)-Cd(1)- Br(4)	107.04 (2)
		Br(3)-Cd(1)-Br(4)	100.81 (2)

Table 3. Intramolecular and intermolecular interactions in the co-crystal $Cd(L)_2Br_4$

D–H···A	r(D–H)/Å	r(D–A)/Å	r(H···A)/Å	∠ D–H···A / °	Symmetry code
C36–H36C···Br1	0.96	3.711(3)	2.754(1)	174.07(2)	-x+1,-y+1,-z+2
C13–H13···Br1	0.93	3.664(4)	2.981(1)	131.53(2)	-x,-y+1,-z+2
C5–H5···Br2	0.93	3.755(3)	2.825(1)	178.37(2)	-x+1,-y+1,-z+2
C25–H25···Br2	0.93	3.828(3)	2.899(1)	176.52(2)	-x,-y+1,-z+2
C22–H22A···Br3	0.96	3.562(3)	2.876(1)	129.29(2)	-x,-y+2,-z+2
C28–H28···Br4	0.93	3.767(2)	2.902(1)	155.28(2)	x+1,+y-1,+z-1
C43–H43···Br4	0.93	3.548(3)	2.908(1)	127.16(2)	x+1,+y-1,+z-1
C44–H44A···Br4	0.96	3.571(4)	2.913(1)	126.79(2)	-x+1,-y,-z+2
C22–H22B···O1	0.96	3.498(4)	2.662(2)	145.70(2)	-x,-y+2,-z+2
C18–H18···O1	0.93	3.496(4)	2.768(2)	135.94(2)	-x,-y+2,-z+2
C27–H27···O3	0.93	3.373(3)	2.815(2)	119.60(2)	x,+y-1,+z

Contd…

C26–H26···O4	0.93	3.566(4)	2.645(2)	170.52(2)	x-1,+y,+z
C15–H15B···O4	0.97	3.686(4)	2.855(2)	144.17(2)	x,y,z
C7–H7···Cg(4)	0.93	3.663(3)	2.95	135	1-X,1-Y,-Z
C17–H17···Cg(3)	0.93	3.819(4)	2.96	155	x,y,z
C36–H36B···Cg(8)	0.96	3.847(4)	2.98	150	X,-1+Y,Z
Cd1–Br3···Cg(1)	2.5144(6)	5.8455(13)	3.7143(14)	139	1-X,1-Y,1-Z
Cg(1)···Cg(2)	-	3.7815(19)	-	-	1-X,1-Y,-Z
Cg(2)···Cg(2)	-	3.9149(19)	-	-	1-X,1-Y,-Z
Cg(5)···Cg(6)	-	3.8580(16)	-	-	1-X,2-Y,1-Z
Cg(6)···Cg(4)	-	3.7135(17)	-	-	X,1+Y,Z

D-donor; A-acceptor; H-hydrogen

Conclusion

The complex $Cd(L)_2Br_4$ has been solved by single crystal x-ray diffraction studies. In the crystal structure, the metal ion, Cd(II) exhibits a distorted tetrahedral coordination of the bromine atoms and is located on a general position.

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