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Synthesis, characterization and coordination properties of 2-(2′-(methylseleno)phenyl)benzimidazole

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ABSTRACT

2-(2′-(methylseleno)phenyl)benzimidazole (**1**) was synthesized from 2-(methylseleno)benzaldehyde and 1,2-diaminobenzene in presence of acetic acid as a catalyst. The reaction of **1** with Pd(II) and Pt(II) afforded square planar complexes **4** and **5** with metal ligand ratio of 1:2. The structures of benzimidazole ligand **1** and complexes **4** and **5** were determined by single crystal X-ray diffraction. Crystal structures showed formation of *cis* isomers and the packing patterns showed formation of supramolecular structures via weak van der Waals interactions. As expected, the hard bases **2** and **3** did not react with soft acids Pd(II) and Pt(II).

1. Introduction

Transition metal coordination compounds containing imidazolic/benzimidazolic ligands have drawn attention of several research groups, since imidazole/benzimidazole and their derivatives are important class of organic compounds in coordination chemistry, photophysics, photochemistry, bioinorganic chemistry and bioorganic chemistry [1]. Esparza-Ruiz et al. have showed the formation of square planar Ni complex with 2-(2′-(thio)phenyl)benzimidazole ligand [2]. The oxoperoxomolybdenum(VI) compound with 2-(2′-hydroxyphenyl)-1H-benzimidazole and ruthenium(II) compounds of 2-(2′-pyridyl)benzimidazole are active against *Entamoeba hystolytica* [3]. Copper(II) and silver(I) compounds of 2-(2′-pyridyl)benzimidazole show activity against *Staphylococcus*, *Pseudomonas aeruginosa*, *Salmonella typhi*, *Shigella flexneri* and *Candida albicans*, while cobalt(II) [4] and platinum(II) [5] compounds are active against *tumor*.

Here we report synthesis of 2-(2′-(methylseleno)phenyl)benzimidazole (**1**) from 2-(methylseleno)benzaldehyde and 1,2-diaminobenzene. The complexation behavior of ligand **1** toward Pd(II) and Pt(II) was studied and compared it with that of sulfur (**2**) [6] and oxygen (**3**) [7] analogues. The reaction of ligand **1** with Pd(II) and Pt(II) afforded the square planar complexes **4** and **5**, respectively, with 1:2 metal to ligand ratio. Benzimidazole ligands were coordinated to the metal center in *cis* fashion.

2. Experimental

2.1. Materials and methods

All reagents were purchased from Aldrich/Merck and used without further purification. Acetonitrile was distilled from P_2O_5 and kept over molecular sieves. 2-methylselenobenzaldehyde [8], $Pd(C_6H_5CN)_2Cl_2$ [8], and $Pt(COD)Cl_2$ [9] were prepared according to the literature reports. UV–Vis spectra were recorded on a Hitachi U4100 spectrometer, with a quartz cuvette (path length, 1 cm). ^{1}H, ^{13}C and ^{77}Se NMR spectra were recorded on a Bruker Avance 500 MHz spectrometer using CD_3CN/DMSO-d_6 as solvent and tetramethylsilane ($SiMe_4$) as internal standard. Data are reported as follows: chemical shifts in ppm (δ). UV–Vis studies were performed in dry acetonitrile. IR spectra of the compounds have been recorded on a Perkin–Elmer spectrum RXI FT-IR spectrometer as KBr pellets. The mass spectra were recorded on a WATERS micromass Q-Tof micro™ instrument. Melting points were measured using a digital melting point apparatus, SECOR INDIA.

2.1.1. 2-(2′-(Methylseleno)phenyl)benzimidazole (1)

2-(Methylseleno)benzaldehyde (582 mg, 2.92 mmol) [8] was refluxed azeotropically in 20 mL dry toluene, with equimolar amount of 1,2-diamminobenzene and two drops of acetic acid, using a Dean–Stark trap. The product was precipitated out from the reaction mixture. White product was filtered and dried under vaccum. Yield: 50% (420 mg, 1.46 mmol). Mp: 178 °C. *Anal.* Calc. for $C_{14}H_{12}N_2Se$: C, 58.54; H, 4.21; N, 9.75. Found: C, 58.75; H, 3.44; N, 10.38%. IR(KBr, cm^{-1}): 3438 cm^{-1} (N–H stretching),

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1432 cm^{-1} ($\nu_{C{=}N}$ stretching); ESI–MS: $C_{14}H_{12}N_2Se + H^+$ (289.0599). ^1H NMR (DMSO-d_6, 500 MHz): δ 12.84 (s, 1H, NH proton), 7.90 (m, 1H), 7.68 (m, 1H), 7.53–7.22 (m, 6H), 2.21 (s, 3H, Me). ^{13}C NMR (DMSO-d_6, 125 MHz): δ 151.30 (N–C–NH), 143.16, 134.85, 134.38, 129.85, 129.14, 128.67, 128.15, 124.80, 122.83, 121.59, 119.00, 111.36 (Ar–C), 6.71 (Se–Me). ^{77}Se NMR (DMSO-d_6, 95 MHz): δ 337 ppm.

*2.1.1.1. 2-(2′-(Methylthio)phenyl)benzimidazole (**2**).* Synthesis of **2** was carried out similar to that of **1** staring from 2-(methylthio)benzaldehyde (480 mg, 3.45 mmol) Yield: 53% (400 mg, 1.67 mmol). ^1H NMR (DMSO-d_6, 500 MHz): δ 12.68 (s, NH proton, 1H), 7.75 (m, 1H), 7.60 (br s, 2H), 7.48 (m, 2H), 7.31(m, 1H), 7.22 (m, 2H), 2.43 (s, Me, 3H).

*2.1.1.2. 2-(2′-(Methoxy)phenyl)benzimidazole (**3**).* Synthesis of **3** was carried out similar to that of **1** starting from 2-methoxybenzaldehyde (523 mg, 3.84 mmol). Yield: 51% (440 mg, 1.96 mmol). ^1H NMR ($CDCl_3$, 500 MHz): δ 10.6 (br s, 1H), 8.49 (m, 1H), 7.54 (br s, 2H), 7.30 (m, 1H), 7.20–7.12 (m, 2H), 7.03 (m, 1H), 6.49 (m, 1H), 3.95 (s, Me, 3H).

2.1.2. General methodology for the preparation of complexes

A mixture of the ligand **1** (50 mg, 0.17 mmol) and $Pd(C_6H_5CN)_2Cl_2$/$Pt(cod)Cl_2$ in 2:1 molar ratio in 10 mL dry methanol was stirred for 3 h at reflux temperature. The reaction mixture was filtered and to the filtrate NH_4PF_6 (28 mg, 0.17 mmol) was added and the product was precipitated, filtered off and dried under vaccum.

*2.1.2.1. Palladium complex **4**.* Yield: 56% (93 mg, 0.096 mmol). Mp: 248 °C, dec. *Anal.* Calc. for $C_{28}H_{24}F_{12}N_4P_2PdSe_2$: C, 34.64; H, 2.49; N, 5.77. Found: C, 34.39; H, 2.13; N, 5.61%. ESI–MS: $C_{28}H_{24}N_4PdSe_2 + H^+$ (683.0055). ^1H NMR (CD_3CN, 500 MHz): δ 12.16 (s, NH proton, 1H), 8.32–6.69 (m, 8H), 2.18 (s, Me, 3H). ^{13}C NMR (CD_3CN, 125 MHz): δ 149.01 (N–C–NH), 140.55, 135.75, 135.05, 133.82, 132.68, 131.87, 126.95, 126.01, 125.87, 123.59, 115.50, 113.93 (Ar–C), 18.60 (Se–Me). ^{77}Se NMR (CD_3CN, 95 MHz): δ 263.05 ppm.

*2.1.2.2. Platinum complex **5**.* Yield 58% (105 mg, 0.10 mmol). Mp: 273 °C, dec. *Anal.* Calc. for $C_{28}H_{24}F_{12}N_4P_2PtSe_2$: C, 31.74; H, 2.28; N, 5.29. Found: C, 31.53; H, 2.07; N, 4.98%. ESI–MS: $C_{28}H_{24}N_4PtSe_2 + H^+$ (772.0858). ^1H NMR (DMSO-d_6, 500 MHz): δ 14.86 (s, NH proton, 1H), 8.43–6.51 (m, 8H), 2.09 (s, Me, 3H). ^{13}C NMR (DMSO-d_6, 125 MHz): δ 146.74 (N–C–NH), 139.14, 134.37, 133.70, 132.79, 132.44, 131.57, 129.70, 125.85, 124.92, 123.49, 115.46, 113.70 (Ar–C), 17.53 (Se–Me). ^{77}Se NMR (CD_3CN, 95 MHz): δ 326.30 ppm.

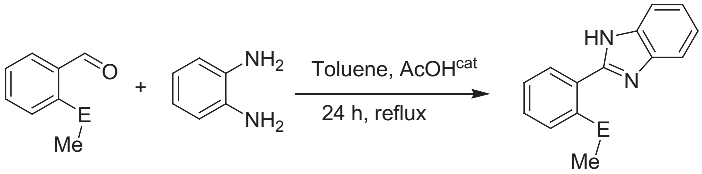
2.2. X-ray crystallography

Single crystals of the compounds suitable for X-ray diffraction were grown from acetonitrile solution by diffusing diethyl ether vapors in a closed beaker. The crystals were carefully chosen using a stereomicroscope supported by a rotatable polarizing stage. The data was collected at RT on Bruker's KAPPA APEX II CCD Duo with graphite monochromated Mo Kα radiation (0.71073 Å). The crystals were glued to a thin glass fibre using FOMBLIN immersion oil and mounted on the diffractometer. The intensity data were processed using Bruker's suite of data processing programs (SAINT), and absorption corrections were applied using SADABS [10]. The crystal structure was solved by direct methods using SHELXS-97 and the data was refined by full matrix least-squares refinement on F^2 with anisotropic displacement parameters for non-H atoms, using SHELXL-97 [11]. Figures are drawn from X-seed version 2.0 [12].

3. Results and discussion

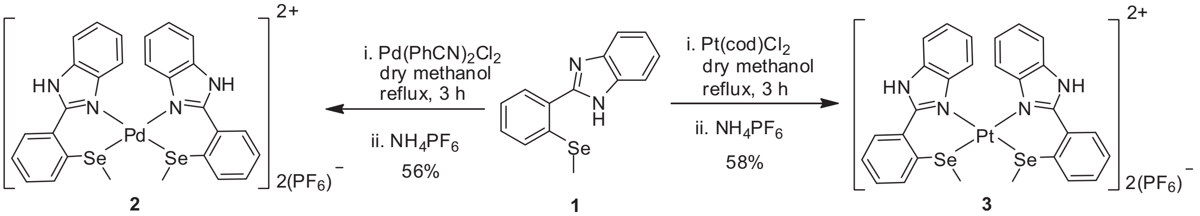
3.1. Ligand Synthesis and characterization

Several reagents [13] have been used as catalyst to prepare the benzimidazole derivatives by the reaction of aldehyde and 1, 2-diaminobenzene. In the synthesis of **1**, acetic acid was used as catalyst [14] for the formation of 2-(2′-(methylseleno)phenyl) benzimidazole. 2-(Methylseleno)benzaldehyde was refluxed azeotropically in toluene with 1,2-diaminebenzene and two drops of acetic acid, using a Dean–Stark trap. 2-(2′-(Methylseleno)phenyl) benzimidazole was precipitated out from reaction mixture as white compound. Thus the acetic acid catalyzed reaction afforded the pure product simply by filtration. Similarly the oxygen and sulfur



1 E = Se (50 %)
2 E = S (53 %)
3 E = O (51 %)

Scheme 1. Synthesis of compound **1–3**.



Scheme 2. Complexation of ligand **1** with Pd(II) and Pt(II).

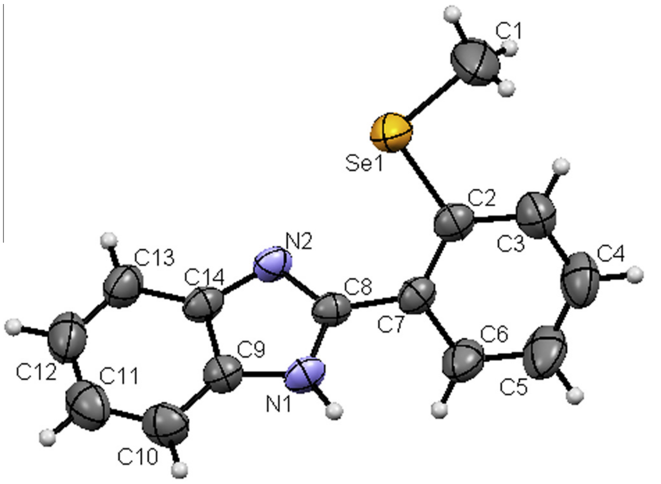


Fig. 1. ORTEP representation of compound **1**; thermal ellipsoids are drawn at 50% probability level.

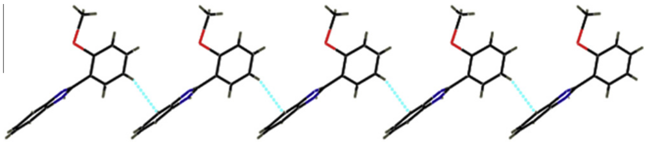


Fig. 2. Formation of 1D chain via C–H···π interactions along *c* axis in the crystal structure of **1**.

analogue were prepared by same strategy using 2-methoxybenzaldehyde and 2-(methylthio)benzaldehyde, respectively (Scheme 1).

The IR spectrum of ligand **1** showed a $\nu_{C=N}$ at $1432\ \mathrm{cm}^{-1}$. Ligand **1** showed a singlet at δ 12.83 ppm for N–H proton in ^{1}H NMR spectrum, whereas for compounds **2** and **3** the peaks appeared at δ 12.68 and δ 10.60 ppm, respectively. The peak at δ 2.20 ppm was assigned to Se–CH_3 in **1** (δ 2.43 ppm for S–Me in **2** and δ 3.95 ppm for O–Me in **3**). In ^{13}C NMR spectrum of ligand **1**, azomethine carbon was observed at δ 151.3 ppm while the peaks arose at δ 150.6 and δ 156.8 ppm for **2** and **3**, respectively. ^{77}Se NMR spectrum showed only one peak at δ 337.0 ppm. Ligand **1** showed the molecular peaks $[(M+H)]^{+}$ at 289.1 as expected in the ESI–MS spectra.

3.2. Complexation study

Ligand **1** was studied for its coordination properties towards Pd(II) and Pt(II). Reactions of ligand **1** with $Pd(C_6H_5CN)_2Cl_2$ and $Pt(COD)Cl_2$, in refluxing methanol, afforded the complexes **4** and **5** where the two benzimidazole moiety coordinated to the metal center through N_2Se_2 to give *cis* isomer. Pd(II) and Pt(II) are soft acids and expected to react with soft base -$SeCH_3$ and are not expected to react with hard base -SCH_3 or -OCH_3.

Reaction of **1** with $Pd(C_6H_5CN)_2Cl_2$ in refluxing methanol for 4 h led to the formation of a yellow solution (Scheme 2). Addition of NH_4PF_6 to the solution afforded yellow powder of complex **4** which was crystallized from acetonitrile/diethyl ether (1:1). Complex **4** is soluble in CH_3CN, DMSO and acetone, however, insoluble in chlorinated solvents. The NMR spectroscopic studies confirmed the formation of selenoether complex **4**. The FT–IR spectrum of **4**

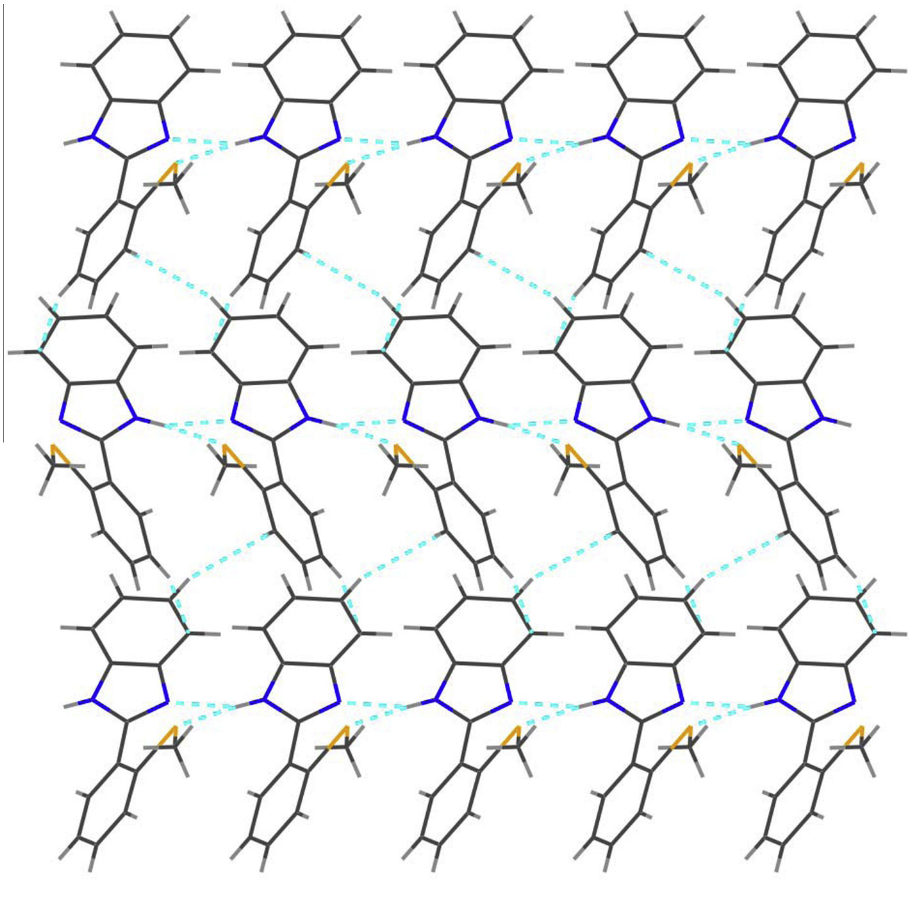


Fig. 3. Formation of 2D sheet packing in the crystal structure of **1** *via* C–H···π, NH···N, NH···Se interactions along *bc*-plane.

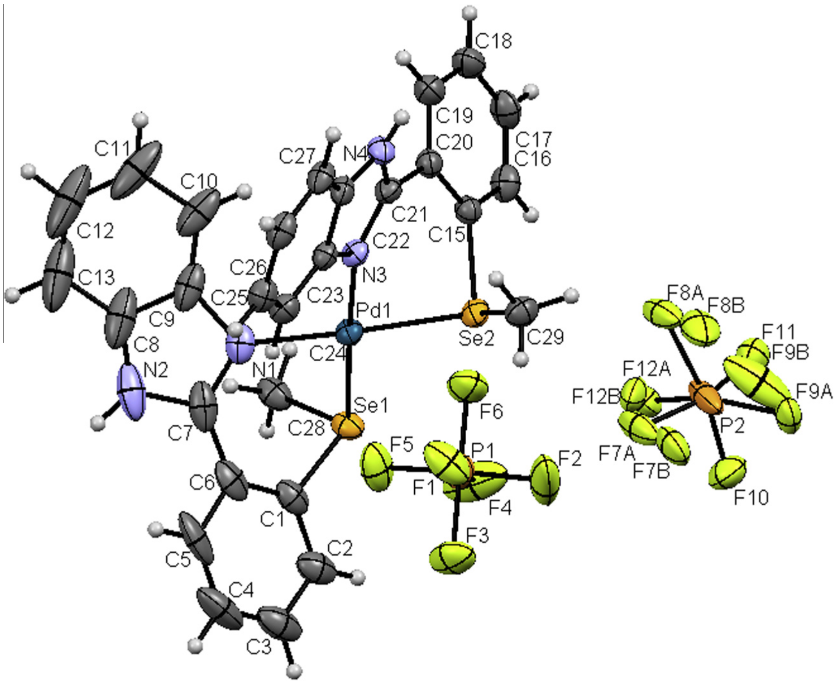


Fig. 4. ORTEP representation of complex **4**; thermal ellipsoids are drawn at 50% probability level.

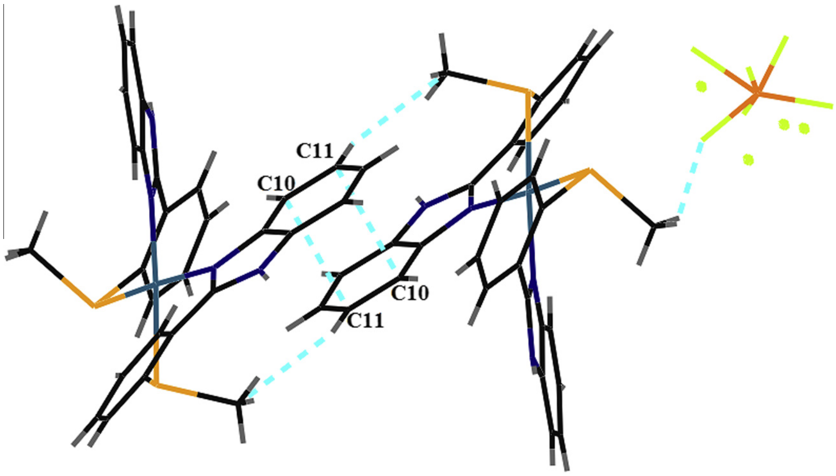


Fig. 5. Intermolecular $\pi \cdots \pi$ interactions present in complex **4**.

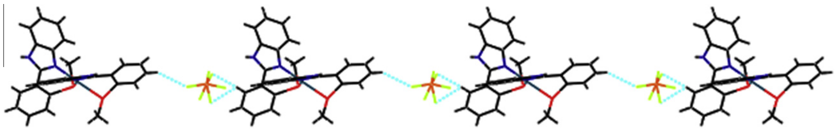


Fig. 6. Formation of 1D chain *via* C–H···F interactions along *a*-axis in the crystal structure of **4**.

indicates the presence of PF_6^- counter ion {846 [ν (P–F)], 558 [δ (F–P–F)] cm^{-1}}. FT–IR spectra displayed the characteristic stretching frequency for imine bond $\nu_{(C=N)}$ at 1440 cm^{-1} which was comparable to that of free ligands **1**. The ^1H NMR spectrum of the complex **4** showed a singlet at δ 12.15 ppm for NH proton which was shifted to upfield compare to that of the ligand **1** (δ 12.83 ppm). The peak at δ 2.93 ppm was assigned to Se–Me fragment in **4** (δ 2.20 ppm for Se–Me in the precursor ligand **1**). ^{77}Se NMR spectrum showed only one peak at δ 263.1 ppm (δ 337.0 ppm for the precursor ligand **1**). The upfield shift is due to co-ordination of Se and N to Pd(II) [15].

Under similar reaction conditions, the reaction of **1** with $Pt(COD)Cl_2$ affforded complex **5**. Complex **5** is soluble in CH_3CN, DMSO and acetone and insoluble in chlorinated solvents. The FT-IR spectrum of **5** indicated the presence of PF_6^- counter ion {845 [ν (P–F)], 559 [δ (F–P–F)] cm^{-1}}. The FT–IR spectrum of **5** displayed

Table 1
Important bond distances (Å) and bond angles (°) in crystal structure of **4**.

S. No	Bond lengths		Bond angles	
1	Pd1–Se1	2.3831(7)	Se1–Pd1–Se2	89.88(2)
2	Pd1–Se2	2.3862(8)	Se1–Pd1–N1	88.89(15)
3	Pd1–N1	2.030(5)	Se1–Pd1–N3	177.28(13)
4	Pd1–N3	2.047(5)	Se2–Pd1–N1	175.19(15)
5	Se1–C1	1.922(6)	Se2–Pd1–N3	87.83(13)
6	Se1–C28	1.913(7)	N1–Pd1–N3	93.5(2)
7	Se2–C29	1.952(6)		
8	Se2–C15	1.929(6)		

the stretching frequency for imine bond $\nu_{(C=N)}$ at 1445 cm^{-1}. The ^{1}H NMR spectrum of the complex **5** showed a singlet at 14.86 ppm for NH proton which was shifted to downfield compare to that of the ligand **1** (δ 12.83 ppm). The peak Se–Me fragment in **5** was appeared at δ 2.08 ppm. ^{77}Se NMR spectrum showed only one peak at 326.3 ppm which is comparable to the ^{77}Se NMR chemical shift of ligand **1**, however, it is significantly downfield compared to that of Pd-complex **4**.

3.3. Crystal structure description

3.3.1. Crystal structure of 2-(2′-(methylseleno)phenyl)benzimidazole (*1*)

Ligand **1** crystallizes in orthorhombic *P*bca space group with one molecule in the asymmentric unit (*Z* = 8, *Z*′ = 1). The ORTEP diagram of the ligand **1** is shown in Fig. 1. The molecules form the 1D chain by C–H···π (H5-C13, 2.758 Å) intermolecular interactions along the *c*-axis as shown in Fig. 2. The molecule has NH···N (H1···N2, 2.024 Å), NH···Se (H1···Se1, 3.095 Å) interaction along b-axis. The interactions present in the molecule leads to the formation of 2D sheet on *bc*-plane showed in the Fig 3.

3.3.2. Crystal structure of palladium complex *4*

Complex **4** crystallizes in the orthorhombic space *P*bca with one molecule and two counter ions in the asymmetric unit (*Z* = 8) with a square planar geometry around the Pd(II) center (Fig. 4). The ORTEP diagram of complex **4** is shown in Fig. 4. The distances between the two Pd–Se bonds (selenoether (Pd1–Se1), 2.3831(7) Å and (Pd1–Se2), 2.3862(8) Å, are very similar and the two Pd–N bond

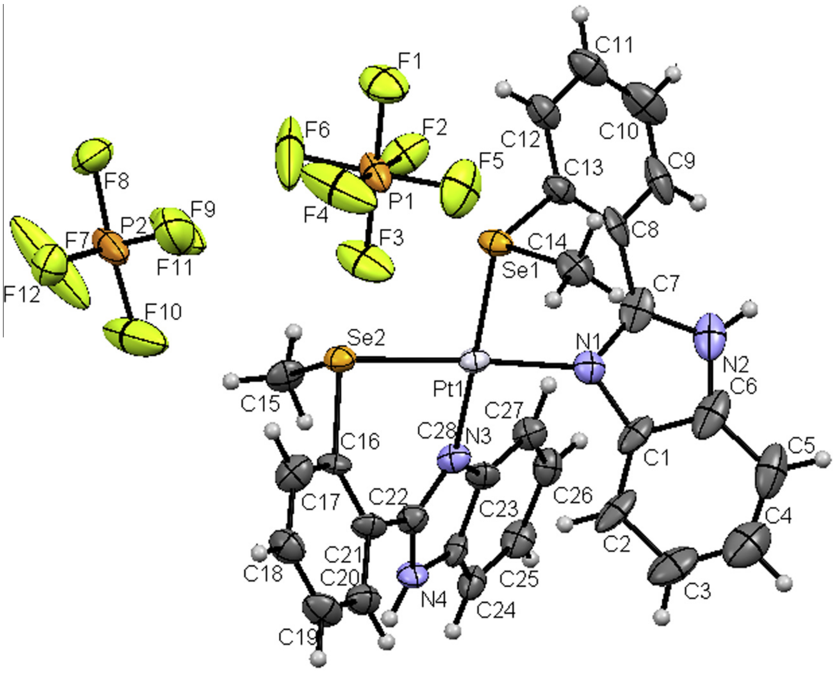


Fig. 7. ORTEP representation of platinum complex **5**; thermal ellipsoids are drawn at 50% probability level.

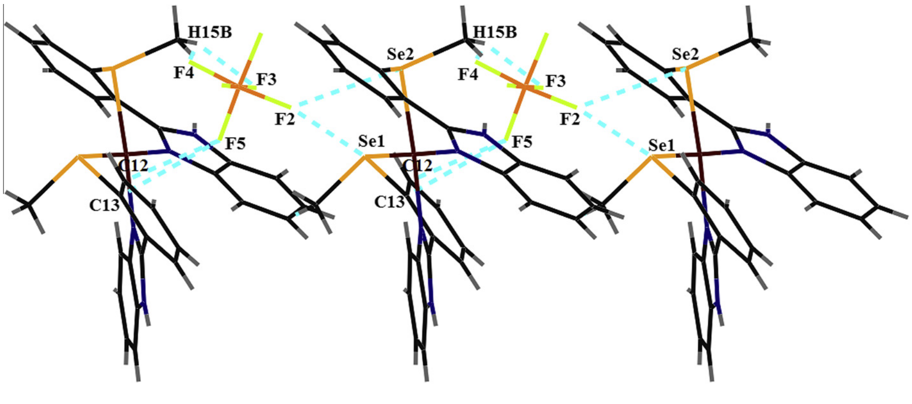


Fig. 8. Crystal packing of complex **5** along *b*-axis through F···Se, H···F, F···π interactions.

Table 2
Important bond distances (Å) and bond angles (°) in crystal structure of **5**.

S. No	Bond lengths		Bond angles	
1	Pt1–Se1	2.3896(11)	Se1–Pt1–Se2	91.77(4)
2	Pt1–Se2	2.3890(12)	Se1–Pt1–N1	89.0(2)
3	Pt1–N1	2.018(8)	Se1–Pt1–N3	177.6(2)
4	Pt1–N3	2.038(8)	Se2–Pt1–N1	175.7(3)
5	Se1–C13	1.925(10)	Se2–Pt1–N3	87.6(2)
6	Se1–C14	1.924(11)	N1–Pt1–N3	91.8(3)
7	Se2–C15	1.959(10)		
8	Se2–C16	1.938(9)		

distances are 2.030(5) and 2.047(5) Å (sum of the covalent radii and van der Waal radii (rvdW) of Pd and N are 2.07 Å, 2.84 Å, respectively). Recently, we have reported bis(methyl)thiosalen Pd(II) complex with the Pd–N distances of 2.023 Å and 2.014 Å [16]. Jain et al. have reported Pd(II) selenolate complex where the Pd–Se distances were found to be of 2.495 Å and 2.479 Å [17]. In one of the PF_6^- anion showed disordered structure. Disorder modeling was failed for rectifying the problem of disorderness. Two benzimidazole ligands coordinate to the Pd(II) center *via cis* fashion. Fig. 5 shows intermolecular $\pi \cdots \pi$ interaction between C10–C11 bonds (3.228 Å). Se⋯F interactions are also present in complex **4** (Se1⋯F1, 3.050 Å, Se2⋯F1, 3.204 Å) (sum of the covalent radii and rvdW of F and Se are 1.77 Å and 3.37 Å respectively). The complex **4** forms the 1D chain by C–H⋯F (H19⋯F7A (2.362 Å), H19⋯F12A (2.618 Å), H3⋯F8A (2.583 Å)) intermolecular interactions along the *a*-axis as shown in Fig. 6. Selected bond lengths and bond angles are shown in Table 1.

*3.3.3. Crystal structure of platinum complex **5***

Complex **5** crystallizes in the orthorhombic space *P*bca with one molecule and two counter ions in the asymmetric unit (*Z* = 8) with a square planar geometry around the Pt(II) center (Fig. 7). The ORTEP diagram of complex **5** is shown in Fig. 7. The two Pt–Se bond distances (selenoether, Pt1–Se1, 2.3896 (11) Å and Pt1–Se2, 2.3890(12) Å), are very similar and the two Pt–N bond distances are 2.038(8) and 2.018(8) Å (sum of the covalent radii and rvdW of Pd and N are 2.04 Å, 2.84 Å, respectively). Like complex **4**, in complex **5** two benzimidazole ligands coordinate to the Pt(II) center to give in *cis* isomer. Molecular structure and packing were found to be very similar to that of complex **4**. Along *b*-axis, complex **5** have F2⋯Se1 (3.044 Å), F2⋯Se2 (3.218 Å), F5⋯π (C_{12}–C_{13}) (3.037 Å), H15B-F3 (2.494 Å), H15B-F4 (2.613 Å) intermolecular interactions (Fig 8). Selected bond lengths and bond angles are shown in Table 2. Complete crystallographic data for ligand **1** and complexes **4** and **5** is provided in Table 3.

4. Conclusion

The reaction of 2-(2′-(methylseleno)phenyl)benzimidazole (**1**) with Pd(II) and Pt(II) afforded square planar complexes **4** and **5**. The structures of benzimidazole ligand **1** and two complexes (**4** and **5**) were determined by single crystal X-ray diffraction analysis. The weak van der Waals interactions lead to the formation of 2D sheet for ligand **1** and 1D chain for the complexes **4** and **5**.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.ica.2013.11.030. CCDC 937556–937558 contains the supplementary crystallographic data for this paper. These data can be obtained free of

Table 3
Crystal data for ligand **1** and complexes **4** and **5**.

	1	4	5
Formula	$C_{14}H_{12}N_2Se$	$C_{28}H_{24}F_{12}N_4P_2Se_2Pd$	$C_{28}H_{24}F_{12}N_4P_2Se_2Pt$
Crystal system	Orthorhombic	Orthorhombic	Orthorhombic
Space group	*P*bca	*P*bca	*P*bca
a [Å]	15.963(3)	17.3412(19)	17.567(3)
b [Å]	9.9018(15)	15.6328(18)	15.691(3)
c [Å]	16.258(3)	23.472(3)	23.742(4)
α [°]	90	90	90
β [°]	90	90	90
γ [°]	90	90	90
V [Å^3]	2569.8(8)	6363.1(13)	6544(2)
Z	8	8	8
λ [Å]	0.71073	0.71073	0.71073
ρ_{calc} [g cm^{-3}]	1.485	2.027	2.183
F[000]	1152	3776	4096
μ [mm^{-1}]	2.901	3.072	6.712
θ [°]	2.51–24.74	2.61–27.51	2.32–24.83
Index ranges	$-20 \leqslant h \leqslant 20$	$-22 \leqslant h \leqslant 22$	$-21 \leqslant h \leqslant 23$
	$-12 \leqslant k \leqslant 12$	$-8 \leqslant k \leqslant 19$	$-20 \leqslant k \leqslant 20$
	$-20 \leqslant l \leqslant 20$	$-29 \leqslant l \leqslant 29$	$-30 \leqslant l \leqslant 31$
T [K]	298(2)	298(2)	298(2)
R_1	0.0444	0.0522	0.0561
wR_2	0.1076	0.1172	0.1169
R_{merge}	0.0707	0.0691	0.1178
Parameters	156	485	453
Goodness-of-fit (GOF)	1.025	0.978	1.035
Reflections total	20871	54542	54302
Unique reflections	2816	6936	7894
Observed reflections	1995	5544	4706
CCDC No.	937557	937556	937558

charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/data_request/cif.

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