

Electronic Supporting Information

Graphene Oxide Grafted with Pd₁₇Se₁₅ Nano-particles Generated from Single Source Precursor as Recyclable and Efficient Catalyst for C–O Coupling in *O*-Arylation at Room Temperature

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1. Experimental Section

General

^1H , $^{13}\text{C}\{^1\text{H}\}$ and $^{77}\text{Se}\{^1\text{H}\}$ NMR spectra were recorded on a Bruker Spectrospin DPX 300 NMR spectrometer at 300.13, 75.47 and 57.24 MHz respectively. The chemical shifts are reported in ppm relative to the residual deuterated solvent or the internal standard (tetramethylsilane in case of ^1H , $^{13}\text{C}\{^1\text{H}\}$ NMR and Me_2Se for $^{77}\text{Se}\{^1\text{H}\}$ NMR). All reactions were carried out in glassware dried in an oven at room temperature. X-ray diffraction data for crystals of **1** was collected on a BRUKER AXS SMART–APEX CCD diffractometer using $\text{Mo-K}\alpha = 0.71073 \text{ \AA}$ radiations. Frames were collected at $T = 298 \text{ K}$ by ω , φ , and 2θ -rotations with full quadrant data collection strategy (four domains each with 600 frames) at 10s per frame with SMART. The measured intensities were reduced to F^2 and corrected for absorption with SADABS.¹ Structure solution, refinement, and data output were carried out with the SHELXTL package by direct methods.² Non-hydrogen atoms were refined anisotropically.

Scanning electron microscope (SEM) studies Carl ZEISS EVO50 instrument was used. Sample was mounted on a circular metallic sample holder with a sticky carbon tape. The associated EDX system Model QuanTax 200, which is based on the SDD technology and provides an energy resolution of 127 eV at Mn K alpha was used for estimating elemental compositions. Powder X-ray diffraction (PXRD) studies were carried out on a Bruker D8 Advance diffractometer using Ni-filtered $\text{CuK}\alpha$ radiation, scan speed of 1 s and scan step of 0.05° . Transmission electron microscopic (TEM) studies were carried out using a JEOL JEM 200CX TEM instrument operated at 200 kV. The specimens for these studies were prepared by dispersing the powdered sample in ethanol by ultrasonic treatment. Few drops of resulting solution were put on a porous carbon film supported on a copper grid and dried in air. Estimation of palladium in nanoparticles was carried out with an AA700 Series atomic absorption spectrometer (Lab India).

Chemicals

Trioctylphosphine (TOP), PdCl_2 , graphite flakes, diphenyldiselenide 1,3-dibromo-2-propanol, sodium borohydride procured from Sigma-Aldrich (USA) were used as received. All the solvents of AR grade i.e. toluene, acetone, chloroform, acetonitrile and ethanol were dried and distilled before use by known standard procedures.³

The synthesis of ligand and complexes are detailed below and summarized in Scheme S1

Synthesis of Ligand L:

Diphenyldiselenide (0.624 g, 2 mmol) dissolved in 30 mL of EtOH was stirred under N₂ atmosphere at room temperature. Sodium borohydride (2 equivalent) was added to it as solid and the mixture was further stirred for 15 min. When it became colourless due to the formation of PhSeNa, 1,3-dibromo-2-propanol (0.436 g, 2 mmol) dissolved in 10 cm³ of ethanol was added with constant stirring and the mixture stirred further for 3 h. It was extracted with chloroform (4 × 25 mL). The extract was washed with water (3 × 40 mL) and dried over anhydrous sodium sulphate. The solvent was evaporated off under reduced pressure on a rotary evaporator to get ligand **L** as yellow oil. Yield (0.59 g, 80%) ¹H NMR (CDCl₃, 25 °C, TMS); (δ, ppm): 2.76 (s, 1H, OH) 3.00 – 3.19 (m, 4H, CH₂Se), 3.81–3.87 (m, 1H, CHOH), 7.23–7.25 (m, 6H, m-Ar, p-Ar), 7.46–7.50 (m, 4H, o-Ar); ¹³C{¹H} NMR (CDCl₃, 25 °C, TMS); (δ, ppm): 34.93 (C₅), 69.12 (C₆), 127.31 (C₁), 129.20 (C₂), 129.26 (C₄), 132.97 (C₃).

Synthesis of Complex 1:

[PdCl₂(CH₃CN)₂] (0.516 g, 0.2 mmol) was added to a stirred solution of **L** (0.074 g, 0.2 mmol) made in CH₃CN (5 mL) at 70° C. The reaction mixture was stirred for 7 h, concentrated with a rotary evaporator and mixed with diethyl ether. The resulting orange coloured compound, was filtered, washed with diethyl ether (10 ml) and dried *in vacuo* to give [Pd(**L**)Cl₂] (**1**) as a yellow solid. Crystals of **1** were grown by slow evaporation of its solution in acetonitrile and methanol mixture (3:1). Yield (0.092 g, 85%), m.p.- 215°C. ¹H NMR (DMSO-d₆, 25 °C, TMS); (δ, ppm): 2.10 (s, 1H, OH), 2.93 – 3.20 (m, 4H, CH₂Se), 3.63 (s, 1H, CHOH), 7.28 – 7.45 (m, 6H, m-Ar, p-Ar), 8.02 (s, 4H, o-Ar); ¹³C{¹H} NMR (DMSO-d₆, 25 °C, TMS); (δ, ppm): 33.74 (C₆), 66.83 (C₅), 128.86 (C₁), 129.76 (C₃), 130.09 (C₄), 133.32 (C₃).

Crystal structure of 1:

The crystals were found long, rod shaped, and orange in colour. The single crystal structure of complex **1** has been solved. Its crystal and refinement data are given in Table T1. In Table T2 bond lengths and angles are given. The geometry of palladium is nearly square planar. The **L** is coordinated with Pd in a bidentate mode forming a six membered chelate ring. The Pd–Se bond lengths of complex **1** are 2.386(14) and 2.389(16) Å,

consistent with earlier reported values⁴ 2.381(7)–2.385(5) Å for species [PdCl₂(Se, N)ligand] and [PdCl(N, Se, Ngand)]⁺, in which also the six membered chelate ring is present.

Synthesis of Graphene Oxide (GO):

GO was synthesized by the modified Hummers' method⁵. In a typical procedure, 500 mg of graphite powder and 2.0 g of sodium nitrate (NaNO₃) were put in cold (below 5 °C) concentrated H₂SO₄ (18 mL, 98%). The mixture was stirred continuously for 1 h and the temperature was kept below 5 °C by cooling in an ice bath. Thereafter, 3 g of potassium permanganate (KMnO₄) was added gradually and reaction was continued for another 2 h at a temperature below 5 °C. The mixture was heated to 35 °C for 30 min and 40 mL of deionised (DI) water was added to it slowly while increasing temperature. It was kept at 100 °C for 15 min, diluted with 70 mL of DI water and cooled to room temperature. The colour of the suspension changed to bright yellow after adding 10 mL of H₂O₂ (35%). The suspension was filtered and washed with 400 mL of 5% HCl twice followed by further washing with 200 mL of DI water for 3 times. Finally, the precipitate was dried in the vacuum desiccator for at least 5 days before further use.

Synthesis of Pd₁₇Se₁₅ Nanoparticles:

A slurry containing 0.1 mmol of complex **1**, in 10 mmol of trioctylphosphine (TOP), prepared in a three necked flask (100 mL) was heated to 100 °C to remove water and oxygen, The resulting homogeneous brown solution was heated to 195 °C under N₂ and kept at the same temperature for 60 min, affording a dark colloidal solution. The solution was air-cooled and NPs of Pd₁₇Se₁₅ were precipitated with excess acetone. They were washed with acetone and dried in air at 60 °C.

Synthesis of Graphene Oxide Grafted with Pd₁₇Se₁₅ Nanoparticles:

In a typical synthesis, 100 mg of GO synthesized as above was completely dissolved in 20 mL of DI water. The 40 mg of Pd₁₇Se₁₅ prepared as above was dispersed in 20 mL of toluene and was added to GO solution. The mixture was stirred for 24 h at room temperature. The precipitate was separated and washed with acetone. The composite so obtained were labelled as GO–Pd₁₇Se₁₅ NPs.

Solubility and Stability of Ligand, Complex and GO-Pd₁₇Se₁₅ NPs:

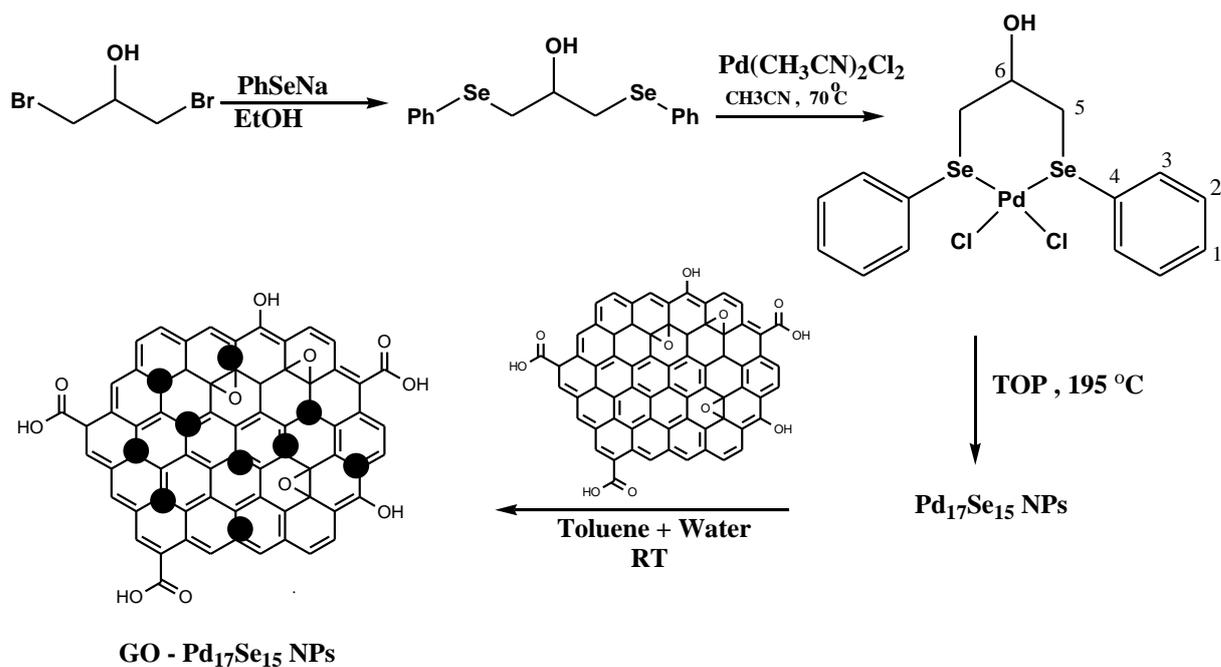
The ligands **L** was found soluble in common organic solvents. The complex **1** shows good solubility in DMF, DMSO, PhCH₃, CH₃CN, CHCl₃ and CH₂Cl₂. It has been found sparingly soluble in THF, and in CH₃OH, diethyl ether and hexane negligibly soluble. On the other hand the catalyst GO- Pd₁₇Se₁₅ NPs have been found sparingly soluble in DMF, DMSO, EtOH and H₂O and insoluble in CHCl₃, CH₃CN, CH₂Cl₂, diethyl ether, hexane and CH₃OH. The solution of catalyst in EtOH becomes turbid within 10-15 min. The ligand as well as its complex **1** may be stored under ambient conditions as they are moisture and air insensitive.

General Procedure for the C–O Coupling Reaction of Aryl/Heteroaryl Halides with Phenol Catalyzed with GO – Pd₁₇Se₁₅ NPs:

An oven-dried flask was charged with aryl halide (1.0 mmol), phenol (1.2 mmol), K₂CO₃ (3.0 mmol) and DMSO (4.0 ml). Catalyst GO–Pd₁₇Se₁₅ NPs (20 mg ;1.0 mol% Pd) in DMSO was added. The reaction was carried out at room temperature under aerobic conditions by stirring the reaction mixture until maximum conversion of aryl halide to product occurred. Thereafter reaction mixture was poured to 50 mL of water. The resulting mixture was extracted with ethylacetate (2 × 50 mL). The organic layer was washed with water (2 × 50 mL) and dried over anhydrous Na₂SO₄. The solvent of the extract was removed with rotary evaporator and the resulting residue was purified by flash column chromatography on silica gel using ethylacetate and hexane mixture as eluent.

Recyclability

To check recyclability of GO–Pd₁₇Se₁₅ NPs , after completion of the C-O cross-coupling of phenol with aryl halides and extraction of cross coupled product with EtOAc the GO–Pd₁₇Se₁₅ NPs present as residue were recovered from the aqueous solution by centrifugation and dried. They were reused for the fresh C-O cross-coupling of phenol with aryl halides.



Scheme S1. Synthesis of Pd(II) complex and Graphene Oxide grafted with $\text{Pd}_{17}\text{Se}_{15}$ NPs

SEM-EDX Data:

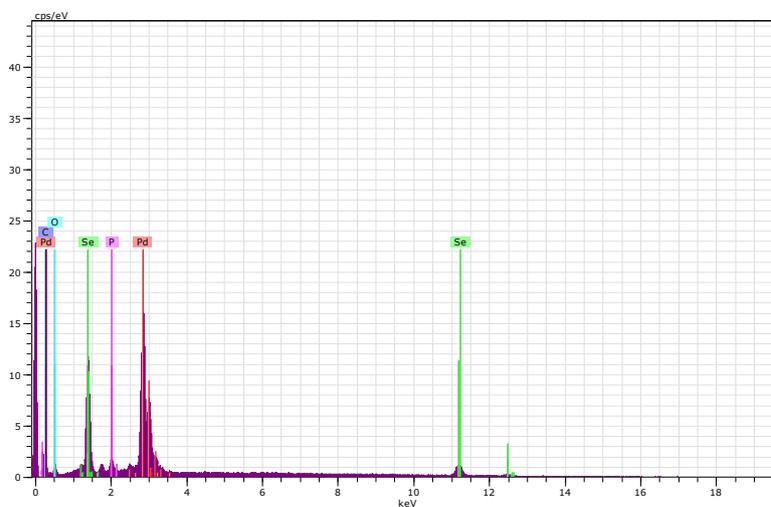


Fig. S1 SEM-EDX of $\text{Pd}_{17}\text{Se}_{15}$ NPs

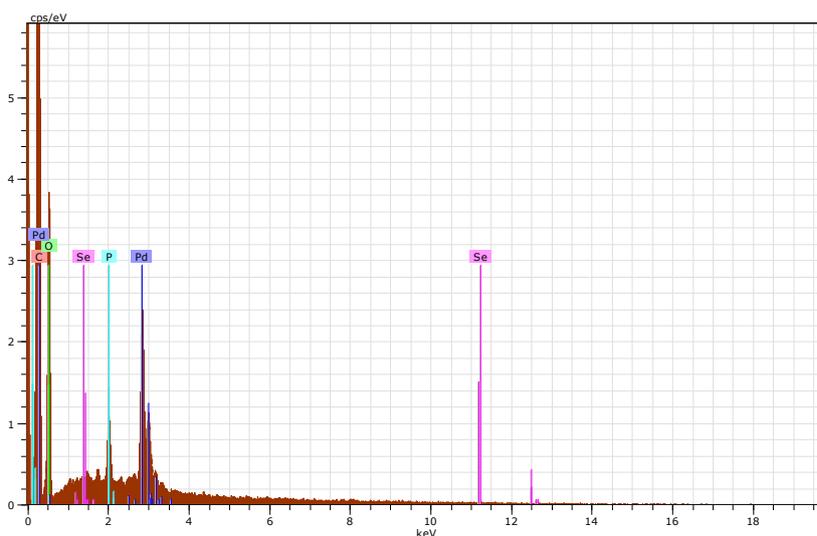


Fig. S2 SEM-EDX of GO-Pd₁₇Se₁₅ NPs

Powder-XRD Data

The powder X-ray diffraction pattern of Pd₁₇Se₁₅ nano-particles (Fig. S4) was indexed on the basis of a primitive cubic unit cell⁶ (JCPDS # 73-1424) with the refined lattice parameter = 10.60 Å and d values (*hkl*): 3.32 (310), 3.17 (311), 2.92 (320), 2.81 (321), 2.56 (410), 2.49 (411), 2.42 (311), 2.36 (420), 2.30 (430), 2.11 (431), 2.06 (511), 2.03 (440), 1.86 (433), 1.76 (600), 1.71 (532), 1.65 (540), 1.63 (541). The diffraction pattern of GO has a peak centered at $2\theta = 11.8^\circ$, corresponding to the [001] interlayer spacing of 7.46 Å.⁷ The XRD pattern of GO-Pd₁₇Se₁₅ NPs (fig. S5) shows clearly diffraction peaks of Pd₁₇Se₁₅, which are without any change in comparison to that of Pd₁₇Se₁₅. However, the [001] reflections of GO are not observed in the XRD pattern of GO-Pd₁₇Se₁₅ NPs sample because the regular stack of GO was destroyed by the intercalation of Pd₁₇Se₁₅ NPs.^{7, 8} The XRD analysis further confirms that Pd₁₇Se₁₅ NPs have been effectively intercalated into the GO stacks.

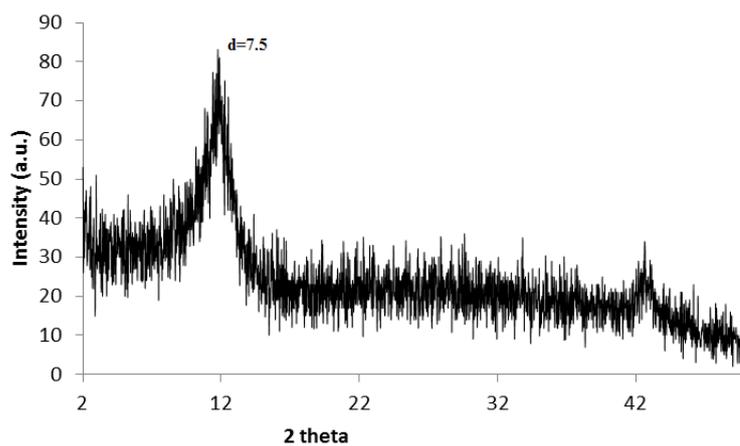


Fig. S3 PXR D of Graphene oxide

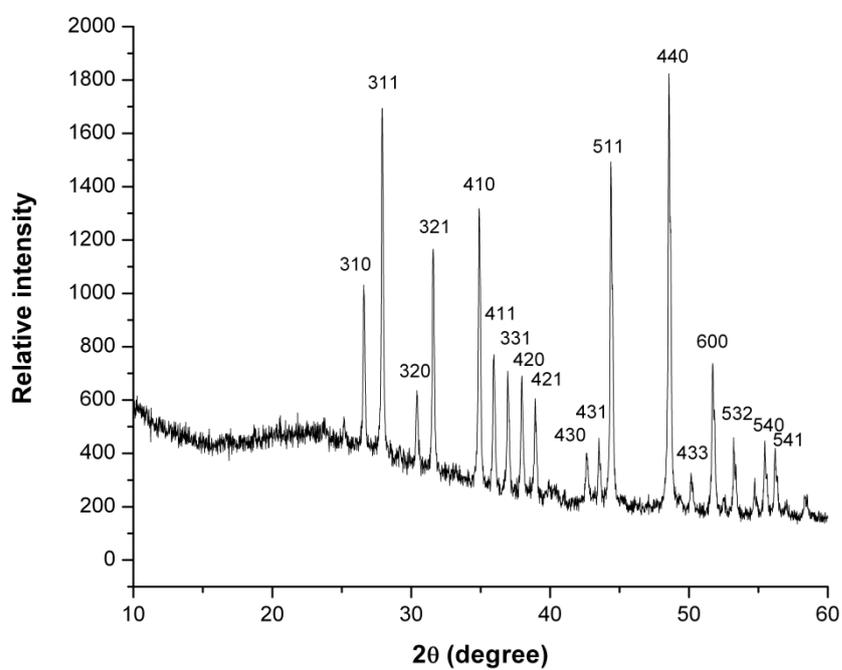


Fig. S4 PXR D of Pd₁₇Se₁₅ NPs

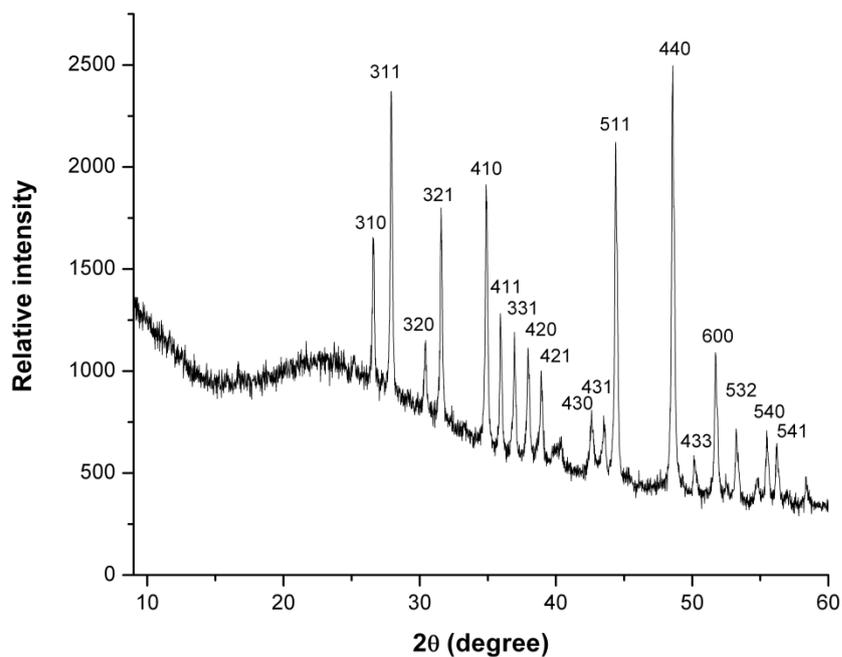


Fig. S5 PXR D of GO-Pd₁₇Se₁₅ NPs

TEM images of GO-Pd₁₇Se₁₅ NPs after 5th Run:

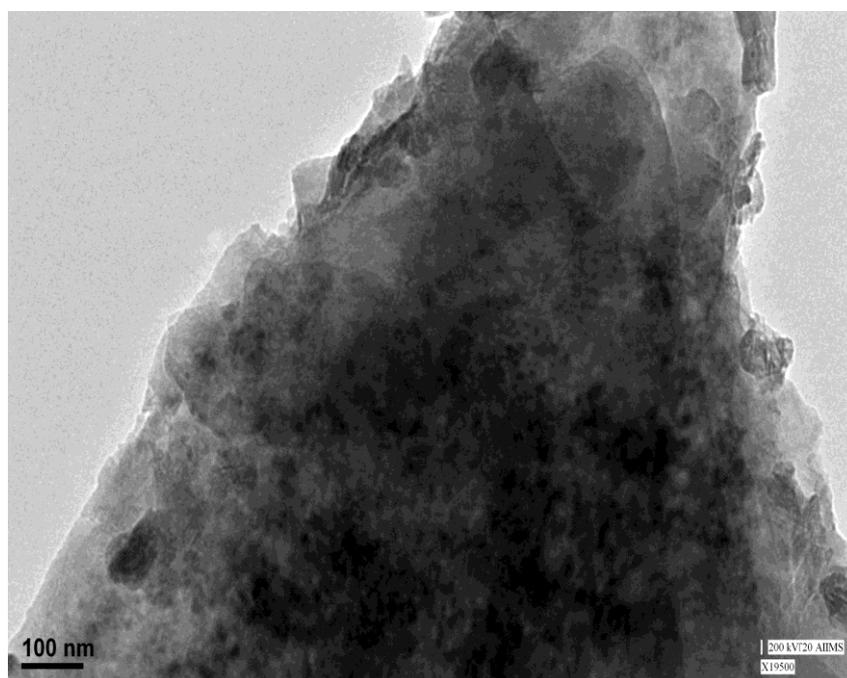


Fig. S6 TEM images of GO-Pd₁₇Se₁₅ NPs after 5th Run at 100 nm scale bar

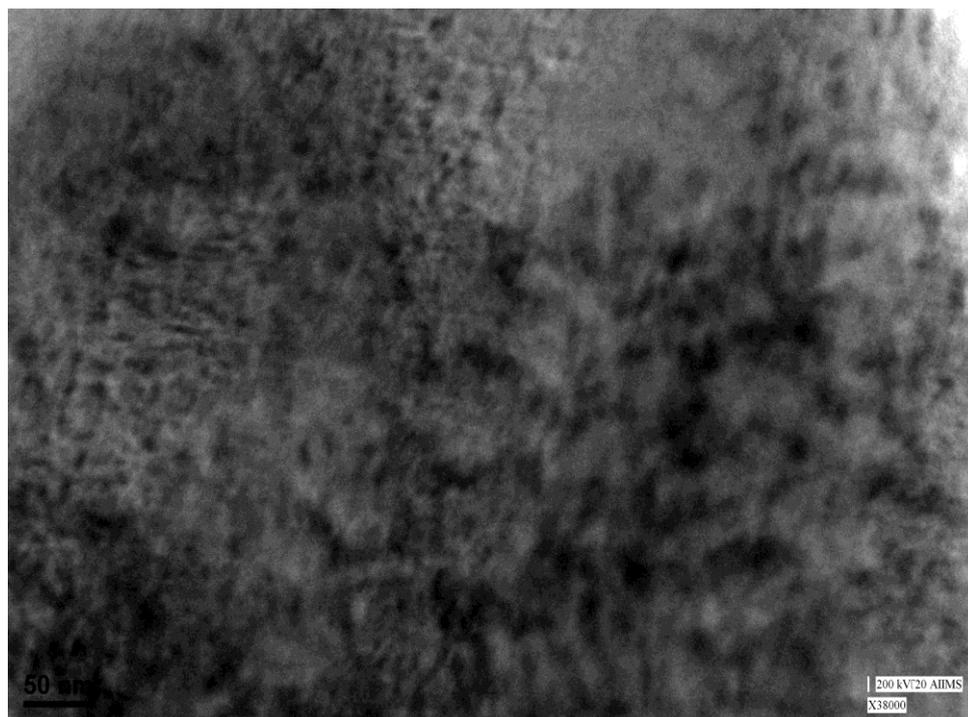


Fig. S7 TEM images of GO-Pd₁₇Se₁₅ NPs after 5th Run at 50 nm scale bar

Crystal data

Table T1. Structural refinement parameters of **1**.

Compounds	1
Empirical formula	C ₁₇ H ₁₉ Cl ₂ N O Pd Se ₂
Formula wt.	588.55
Crystal size [mm]	0.35×0.24×0.23
Crystal system	Monoclinic
Space group	<i>P2(1)/n</i>
Unit Cell dimension	$a = 15.652(9)\text{Å}$ $b = 08.153(5)\text{Å}$ $c = 16.180(10)\text{Å}$ $\alpha = 90.00^\circ$ $\beta = 94.451(10)^\circ$ $\gamma = 90.00^\circ$

Volume [\AA^3]	2058(2)
Z	4
Density (Calc.) [$\text{Mg}\cdot\text{m}^{-3}$]	1.899
Absorption coeff. [mm^{-1}]	4.700
$F(000)$	1136.0
θ range [$^\circ$]	2.53–24.47
Index ranges	$-18 \leq h \leq 18$ $-09 \leq k \leq 09$ $-19 \leq l \leq 19$
Reflections collected	15028
Independent reflections ($R_{\text{int.}}$)	3462 (0.0793)
Max./min. Transmission	0.342/0.226
Data/restraints/parameters	3462/0/219
Goodness-of-fit on F^2	0.930
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0524,$ $wR_2 = 0.1210$
R indices (all data)	$R_1 = 0.0784,$ $wR_2 = 0.1344$
Largest diff. peak/hole [$\text{e}\cdot\text{\AA}^{-3}$]	1.090/–1.570

Table T2. Selected bond lengths [\AA] and bond angles [$^\circ$] of complex **1**

Bond length [\AA]	Bond angle [$^\circ$]
Pd(1)—Se(1) 2.389(16)	Se(1)—Pd(1)—Se(2) 101.05(3)
Pd(1)—Se(2) 2.386(14)	Se(1)—Pd(1)—Cl(1) 84.21(6)
Pd(1)—Cl(1) 2.327(2)	Se(1)—Pd(1)—Cl(2) 174.46(6)
Pd(1)—Cl(2) 2.321(2)	Se(2)—Pd(1)—Cl(1) 174.29(6)
O(1)—C(8) 1.450(8)	Se(2)—Pd(1)—Cl(2) 82.58(6)
Se(1)—C(6) 1.943(7)	Cl(1)—Pd(1)—Cl(2) 92.34(8)
Se(1)—C(7) 1.925(7)	C(7)—C(8)—O(1) 105.2(6)
Se(2)—C(9) 1.961(7)	C(9)—C(8)—O(1) 106.5(6)
Se(2)—C(10) 1.936(8)	C(6)—Se(1)—C(7) 99.6(3)
C(7)—C(8) 1.504(10)	C(9)—Se(2)—C(10) 96.7(3)
C(8)—C(9) 1.510(10)	C(1)—C(6)—Se(1) 122.4(6)
	C(11)—C(10)—Se(2) 124.6(6)

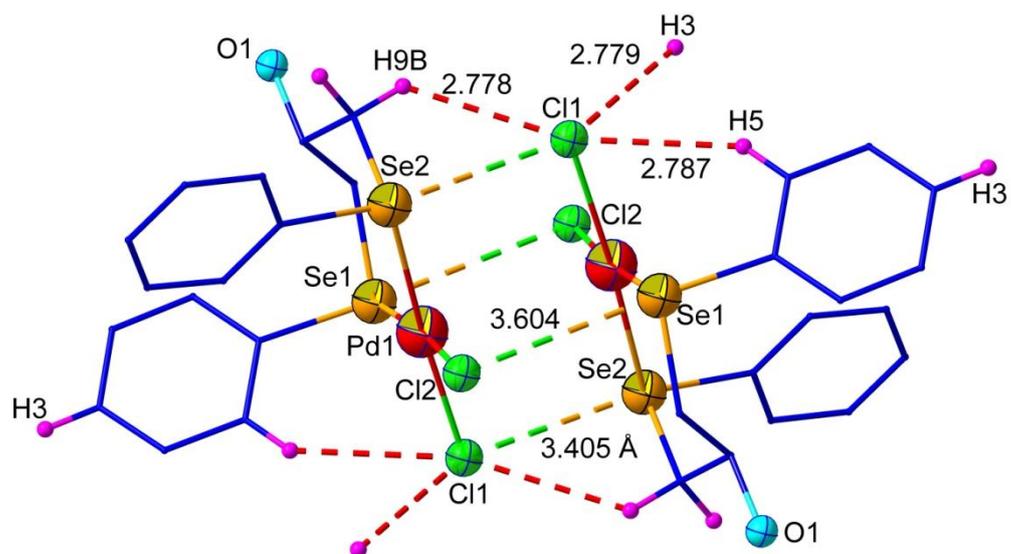


Figure S7. Inter and intra-molecular $\text{Cl} \cdots \text{H}-\text{C}$ and $\text{Cl} \cdots \text{Se}$ interactions in **1**.

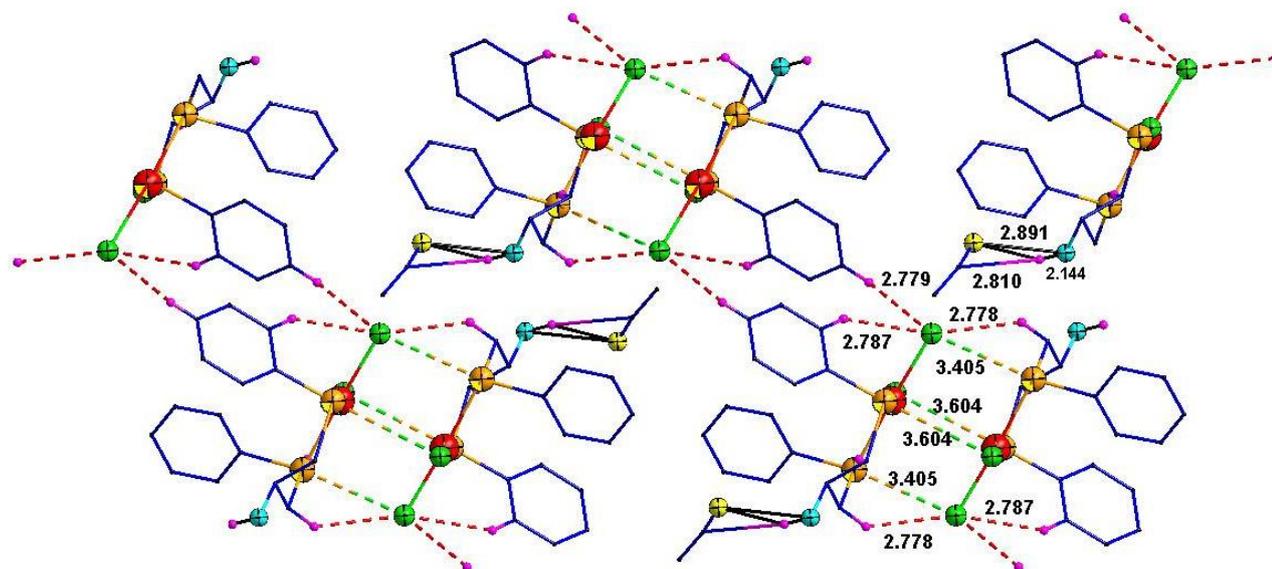
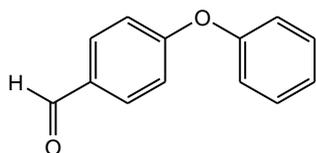
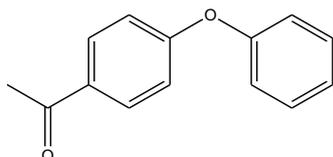


Figure S8. Inter and intra-molecular $\text{Cl} \cdots \text{H}$, $\text{Cl} \cdots \text{Se}$ and $\text{OH} \cdots \text{CH}_3\text{CN}$ interactions in **1**.



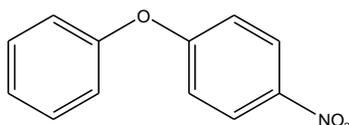
4-phenoxybenzaldehyde:⁹

Yellow liquid. ¹H NMR (300 MHz, CDCl₃): 9.92 (s, 1H), 7.84-7.86 (d, 2H), 7.40-7.45 (t, 2H), 7.20-7.26 (m, 1H), 7.05-7.11 (t, 4H).



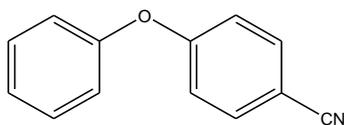
1-(4-phenoxyphenyl)ethanone:⁹

White Solid. ¹H NMR (300 MHz, CDCl₃): 7.91-7.96 (d, 2H), 7.36-7.42 (t, 2H), 7.19-7.22 (t, 1H), 7.06-7.08 (d, 2H), 6.97-7.02 (d, 2H), 2.57 (s, 3H).



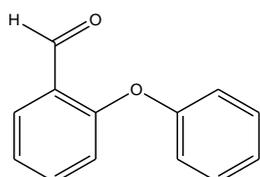
1-Nitro-4-phenoxybenzene:¹⁰

Yellow Solid. ¹H NMR (300 MHz, CDCl₃): 8.11-8.14 (d, 2H), 7.34-7.40 (t, 2H), 7.16-7.21 (t, 1H), 7.01-7.03 (d, 2H), 6.92-6.95 (d, 2H).



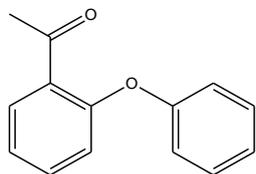
4-phenoxybenzotrile:⁹

White Solid. ¹H NMR (300 MHz, CDCl₃): 7.49-7.55 (m, 2H), 7.30-7.35 (t, 2H), 7.11-7.17 (t, 1H), 6.96-6.98 (d, 2H), 6.90-6.93 (d, 2H).



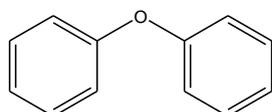
2-phenoxybenzaldehyde:⁹

Yellow liquid. ¹H NMR (300 MHz, CDCl₃): 10.45 (s, 1H), 7.85-7.88 (d, 1H), 7.41-7.47 (t, 1H), 7.30-7.35 (t, 2H), 7.10-7.14 (t, 2H), 6.98-7.01 (d, 2H), 6.81-6.84 (d, 1H).



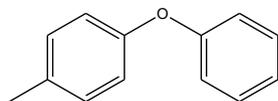
1-(2-phenoxyphenyl)ethanone:¹⁰

Colorless oil. ¹H NMR (300 MHz, CDCl₃): 7.83-7.86 (d, 1H), 7.35-7.45 (m, 3H), 7.15-7.20 (m, 2H), 7.01-7.03 (d, 2H) 6.90-6.92 (d, 2H) 2.64 (s, 3H).



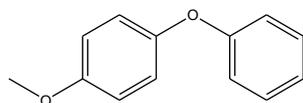
Diphenyl ether:¹⁰

Colorless liquid. ¹H NMR (300 MHz, CDCl₃): 7.24-7.28 (t, 4H), 7.02-7.05 (t, 2H), 6.93-6.95 (d, 4H).



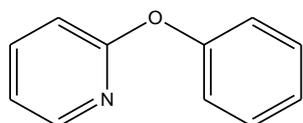
1-Methyl-4-phenoxybenzene:¹⁰

Colorless liquid. ¹H NMR (300 MHz, CDCl₃): 7.46-7.48 (m, 2H), 7.23-7.26 (t, 2H), 7.06-7.07 (m, 1H), 6.98-7.04 (m, 2H), 6.89-6.91 (m, 2H), 2.26 (s, 3H).



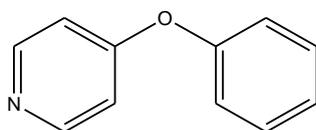
1-Methoxy-4-phenoxybenzene:⁹

Colorless liquid. ¹H NMR (300 MHz, CDCl₃): 7.25-7.32 (m, 2H), 7.01-7.06 (t, 1H), 6.93-7.01 (m, 4H), 6.86-6.90 (m, 2H), 3.81 (s, 3H).



2-phenoxy pyridine:¹⁰

Colorless oil. ¹H NMR (300 MHz, CDCl₃): 8.12-8.13 (m, 1H), 7.58-7.62 (t, 1H), 7.31-7.35 (t, 2H), 7.05-7.18 (m, 3H), 6.90-6.93 (t, 1H), 6.81-6.84 (d, 1H).

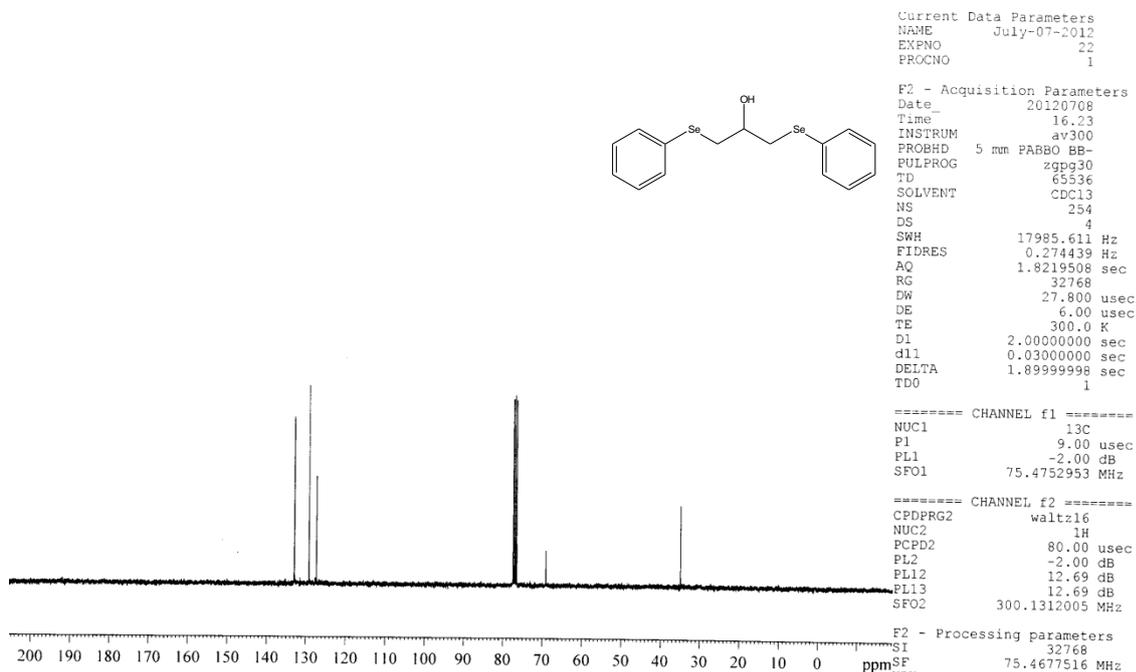
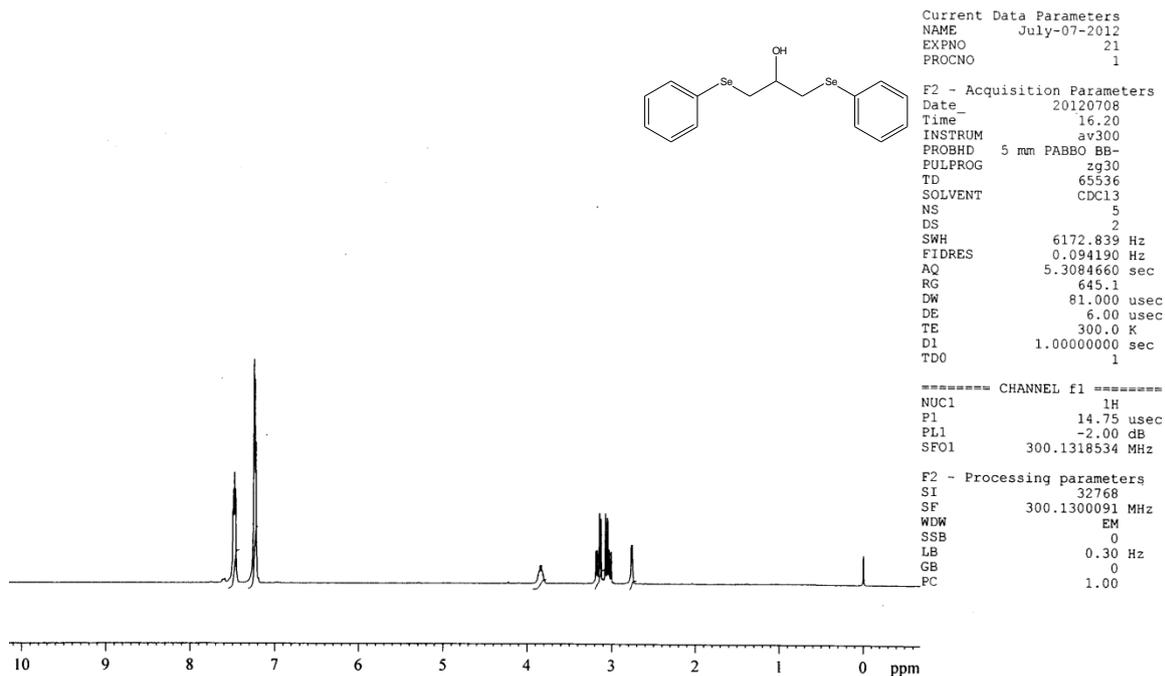


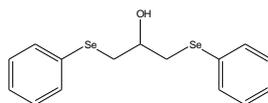
4-phenoxy pyridine:¹¹

Colorless oil. ¹H NMR (300 MHz, CDCl₃): 8.45-8.47 (d, 2H), 7.41-7.47 (t, 2H), 7.20-7.26 (m, 1H), 7.09-7.12 (d, 2H), 6.85-6.91 (m, 2H).

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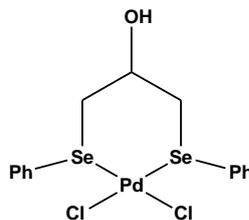
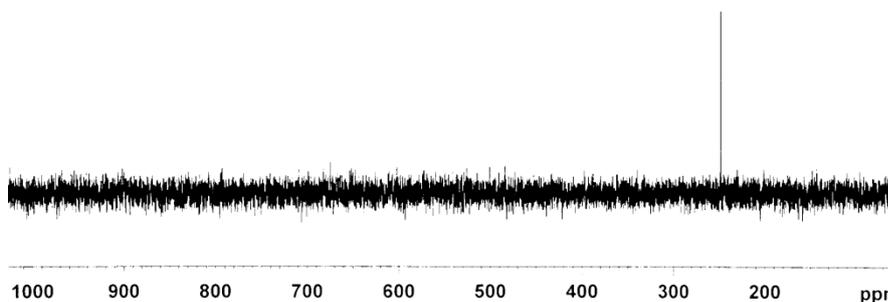


```

===== CHANNEL f1 =====
NUC1      77Se
PC1       10.00 usec
PL1       -2.00 dB
SFO1     51.2708140 MHz

===== CHANNEL f2 =====
PROGPRG  waltz16
NUC1      1H
PC1P1    80.00 usec
PL1P1    -1.00 dB
PL1P2    16.00 dB
SFO1     300.1310000 MHz

F2 - Processing parameters
SI        32768
SF        300.1300010 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



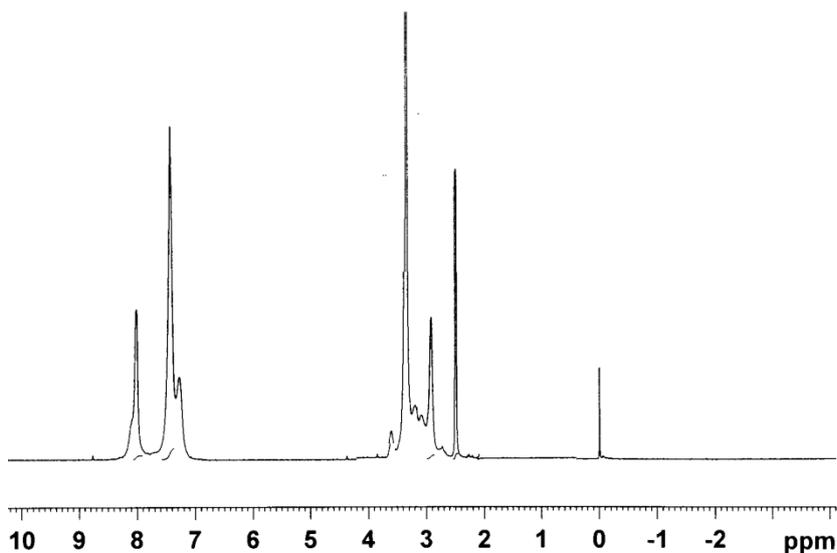
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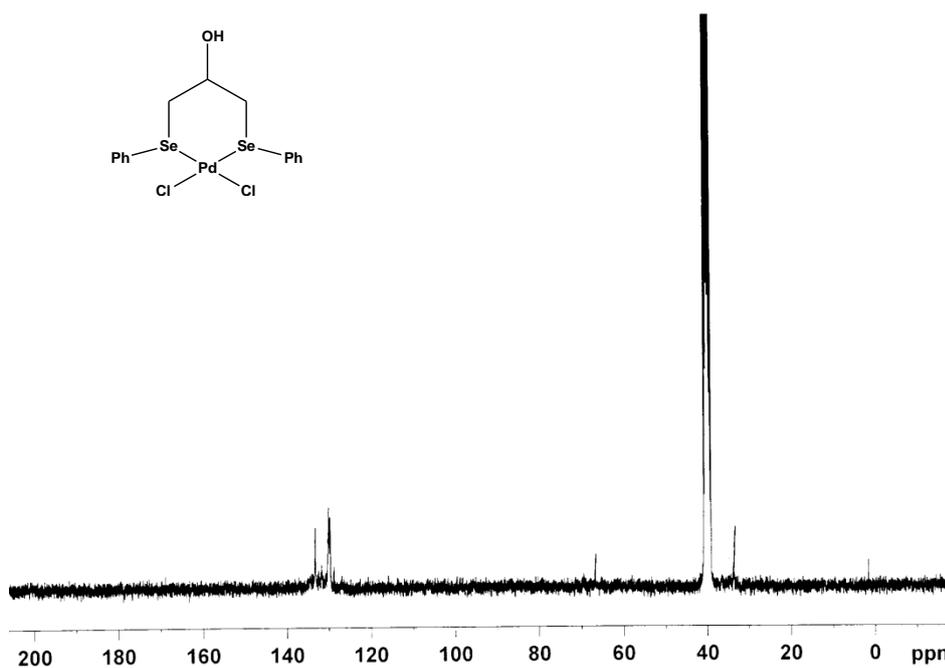
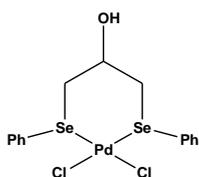
Current Data Parameters
NAME      om
EXPNO    60
PROCNO   1

F2 - Acquisition Parameters
Date_    20110828
Time_    22.29
INSTRUM  av300
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  DMSO
NS       40
DS       0
SWH      6172.839 Hz
FIDRES   0.094190 Hz
AQ       5.3084660 sec
RG       203.2
DW       81.000 usec
DE       6.00 usec
TE       300.0 K
D1       1.00000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1      1H
P1        10.70 usec
PL1       -2.00 dB
SFO1     300.1318534 MHz

F2 - Processing parameters
SI        32768
SF        300.1300010 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



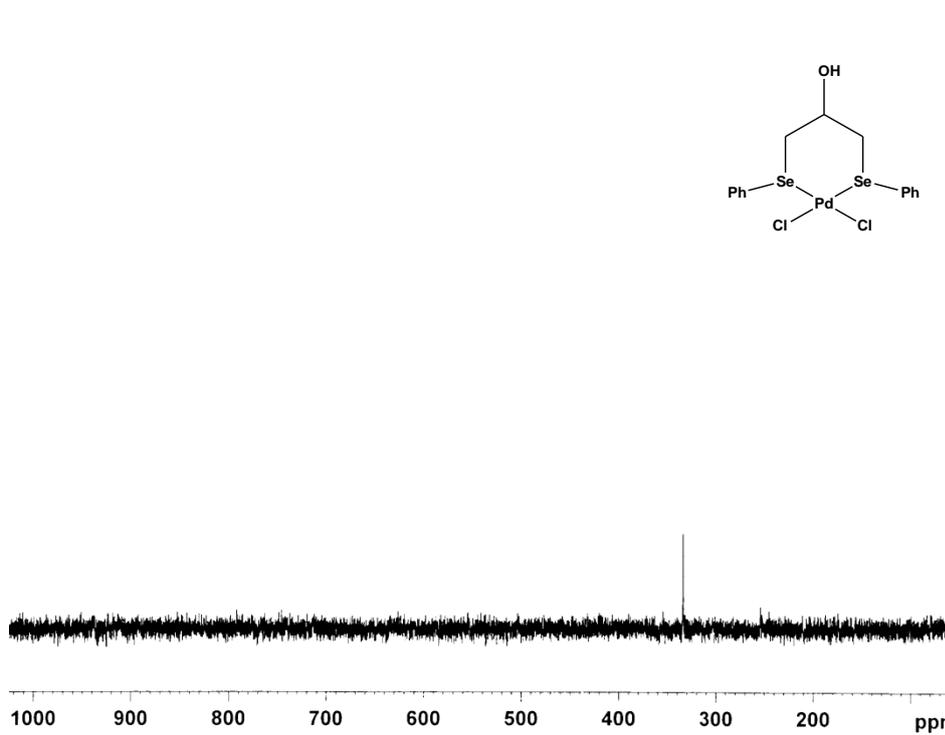
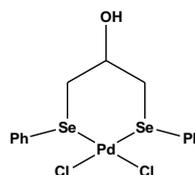


```

===== CHANNEL f1 =====
NUC1      13C
P1         8.30 usec
PL1        -2.00 dB
SFO1      75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2   wait:16
NUC2       1H
PCPD2     80.00 usec
PL2        -2.00 dB
PL12       15.80 dB
PL13       19.80 dB
SFO2      300.1312005 MHz

===== Processing parameters =====
SI         32768
SF         75.4677490 MHz
WDW        EM
SSB         0
GB         1.00 Hz
    
```



```

===== CHANNEL f1 =====
NUC1      79Se
P1         10.00 usec
PL1        -2.00 dB
SFO1      57.2706140 MHz

===== CHANNEL f2 =====
CPDPRG2   wait:16
NUC2       1H
PCPD2     80.00 usec
PL2        -1.00 dB
PL12       16.23 dB
SFO2      300.1312000 MHz

===== Processing parameters =====
SI         32768
SF         57.2703325 MHz
WDW        EM
SSB         0
GB         4.00 Hz
LB         0
GB         0
    
```