

Electronic Supporting Information

Deposition of MoSe₂ flakes using cyclic selenides

Jaroslav Charvot,^a Raul Zazpe,^{b,c} Richard Krumpolec,^d Jhonatan Rodriguez-Pereira,^{b,c} David Pavliňák,^d Daniel Pokorný,^a Milan Klikar,^a Veronika Jelínková,^e
Jan M. Macak^{b,c} and Filip Bureš^{*a,e}

^aInstitute of Organic Chemistry and Technology, Faculty of Chemical Technology, University of Pardubice, Studentská 573, Pardubice, 53210, Czech Republic.

^bCenter of Materials and Nanotechnologies, Faculty of Chemical Technology, University of Pardubice, Nám. Čs. Legií 565, Pardubice, 53002, Czech Republic.

^cCentral European Institute of Technology, Brno University of Technology, Purkyňova 123, Brno, 61200, Czech Republic.

^dDepartment of Physical Electronics, CEPLANT — R&D Center for Plasma and Nanotechnology Surface Modifications, Faculty of Science, Masaryk University, Kotlářská 267/2, 61137 Brno, Czech Republic

^eThe Institute of Technology and Business in České Budějovice, Okružní 517/10, 370 01, České Budějovice, Czech Republic

Table of contents

1. Synthesis.....	2
1.2. Attempted preparation of 2,2,4,4-tetramethyl-1,3,2,4-diselenadisiletane.....	2
2. Nuclear magnetic resonance spectroscopy.....	3
2.1. ¹ H NMR spectra	3
2.2. ¹³ C NMR spectra	5
2.3. ²⁹ Si NMR spectra.....	7
2.4. ⁷⁷ Se NMR spectra.....	9
3. GC/MS records	11
4. DSC thermograms.....	15
5. TGA.....	17
6. SEM.....	19
7. Raman spectroscopy	20
8. XPS.....	21

1. Synthesis

1.2. Attempted preparation of 2,2,4,4-tetramethyl-1,3,2,4-diselenadisiletane

The title compound was attempted from Me_2SiCl_2 (1.6 ml, 1.6 g, 12.6 mmol) following the Methods A or B as well as from MeSiHCl (1.4 ml, 1.2 g, 12.6 mmol) following the Method C. The crude product is a yellow oil. All three methods provided similar results. Figure S1 shows GC/MS records of the crude reaction mixture using Method C at 100 and 250 °C. Three main products **A–C** were identified but the reproducibility of these experiments was rather low. The title compound (**A**) was identified in the crude reaction mixture but all attempts on its purification, including vacuum distillation, crystallization at -78°C and sublimation, failed.

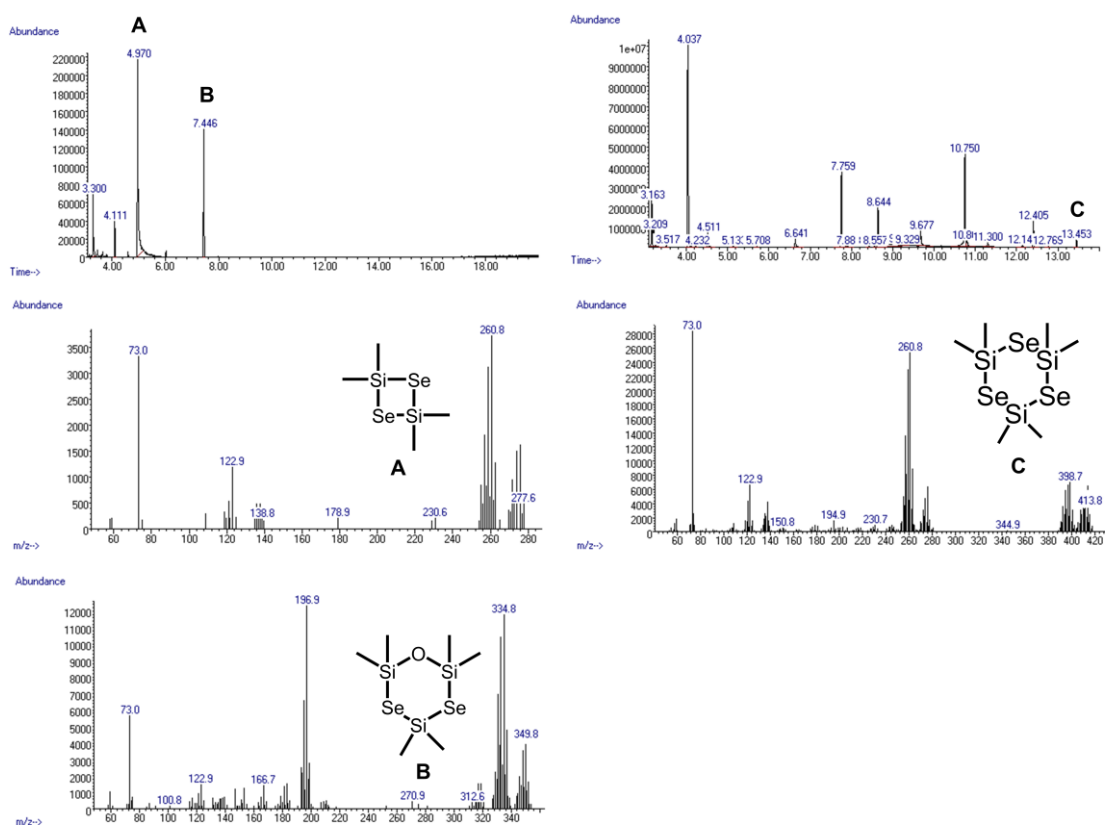


Figure S1 GC/MS record of crude reaction mixture during attempted preparation of 2,2,4,4-tetramethyl-1,3,2,4-diselenadisiletane. Method C at 100 (left) and 250 °C (right).

2. Nuclear magnetic resonance spectroscopy

2.1. ^1H NMR spectra

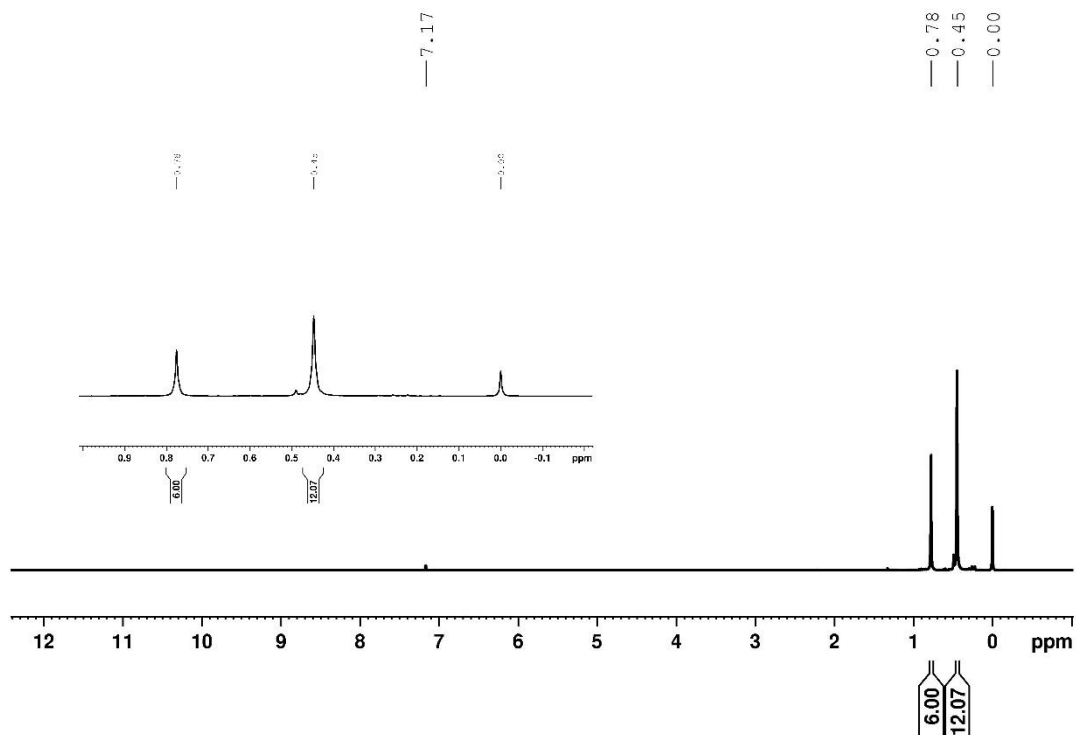


Figure S2 ^1H -NMR (400 MHz, 25 $^\circ\text{C}$, C_6D_6) spectra of 4.

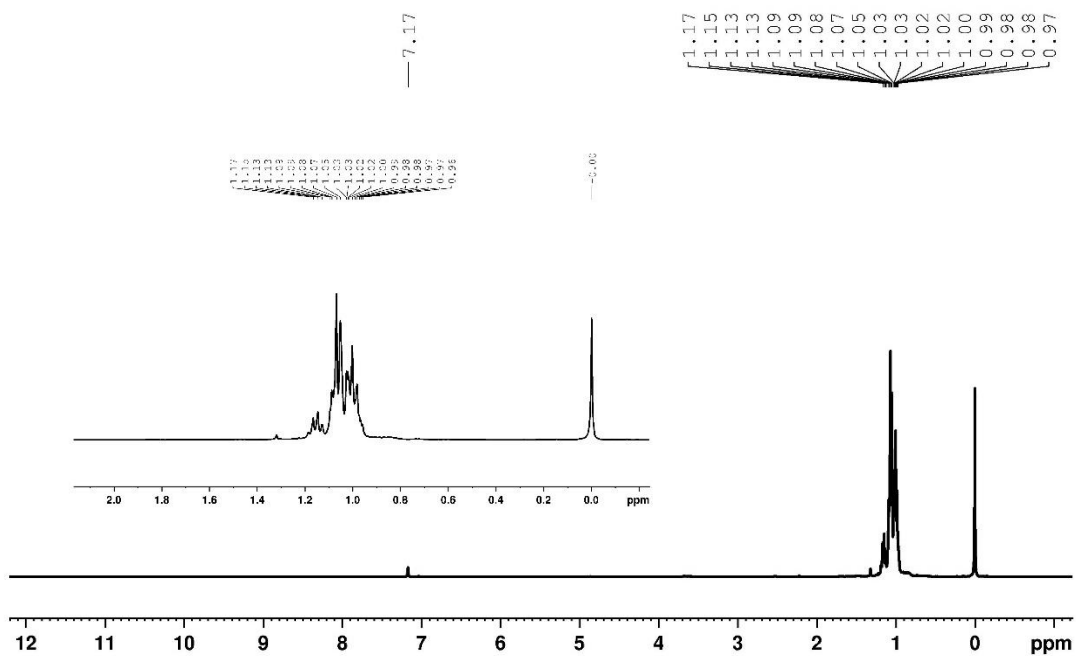


Figure S3 ^1H -NMR (400 MHz, 25 $^\circ\text{C}$, C_6D_6) spectra of 5 (mixture).

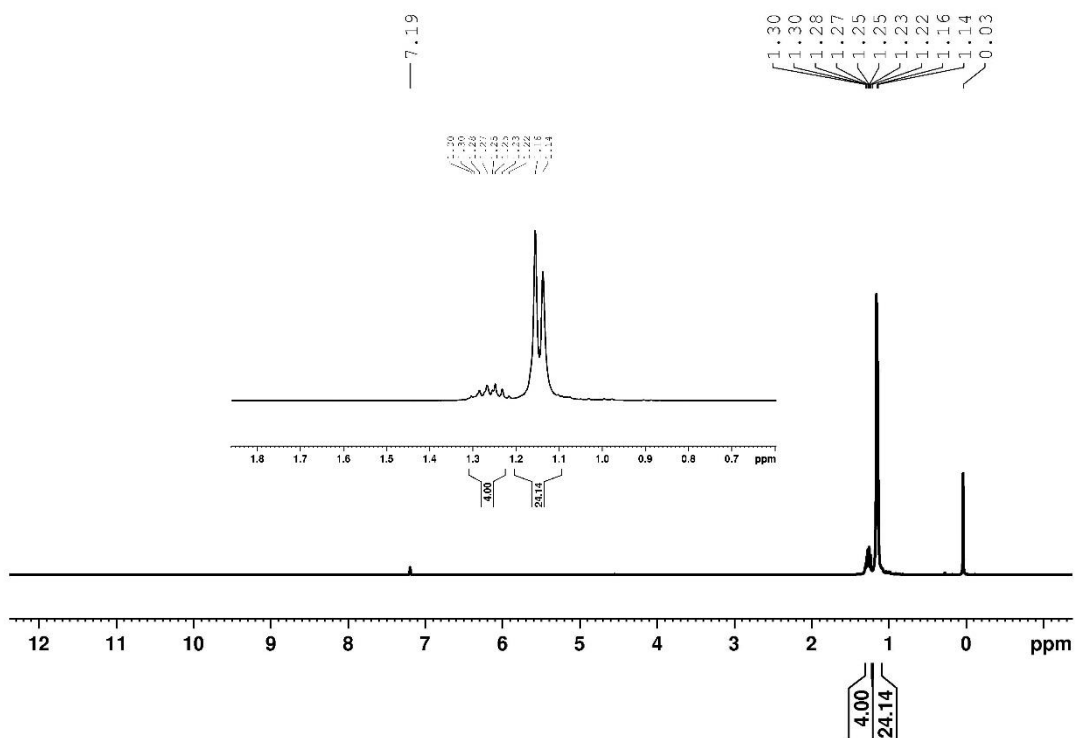


Figure S4 $^1\text{H-NMR}$ (400 MHz, 25 $^\circ\text{C}$, C_6D_6) spectra of **6**.

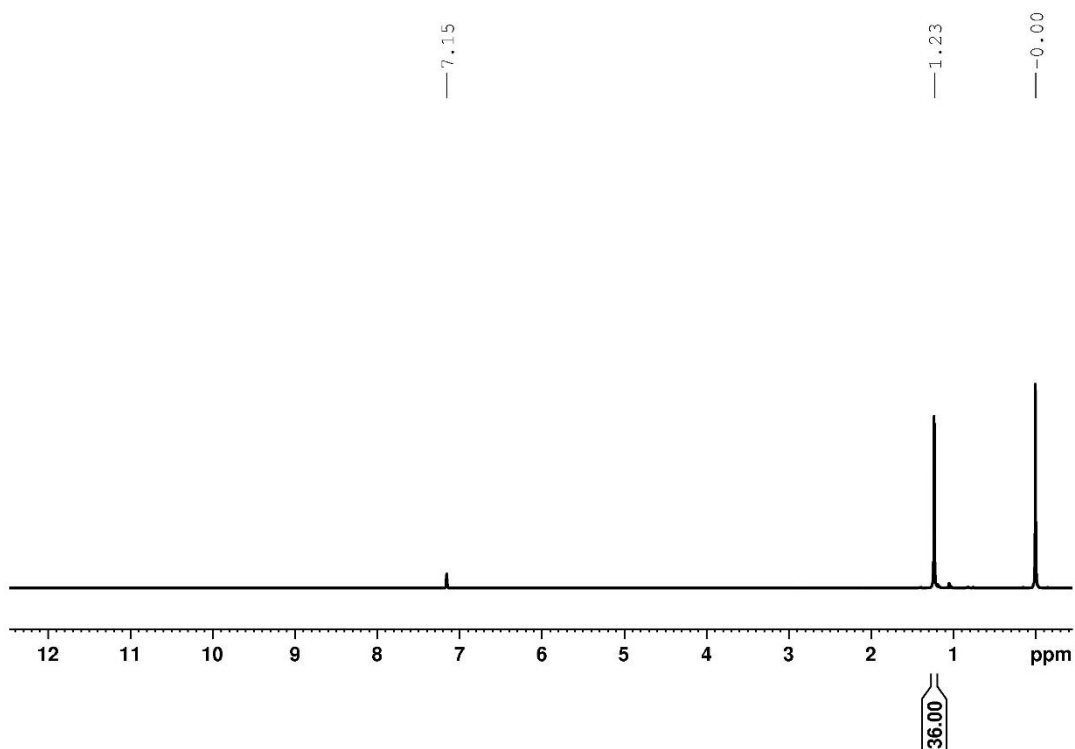


Figure S5 $^1\text{H-NMR}$ (400 MHz, 25 $^\circ\text{C}$, C_6D_6) spectra of **7**.

2.2. ^{13}C NMR spectra

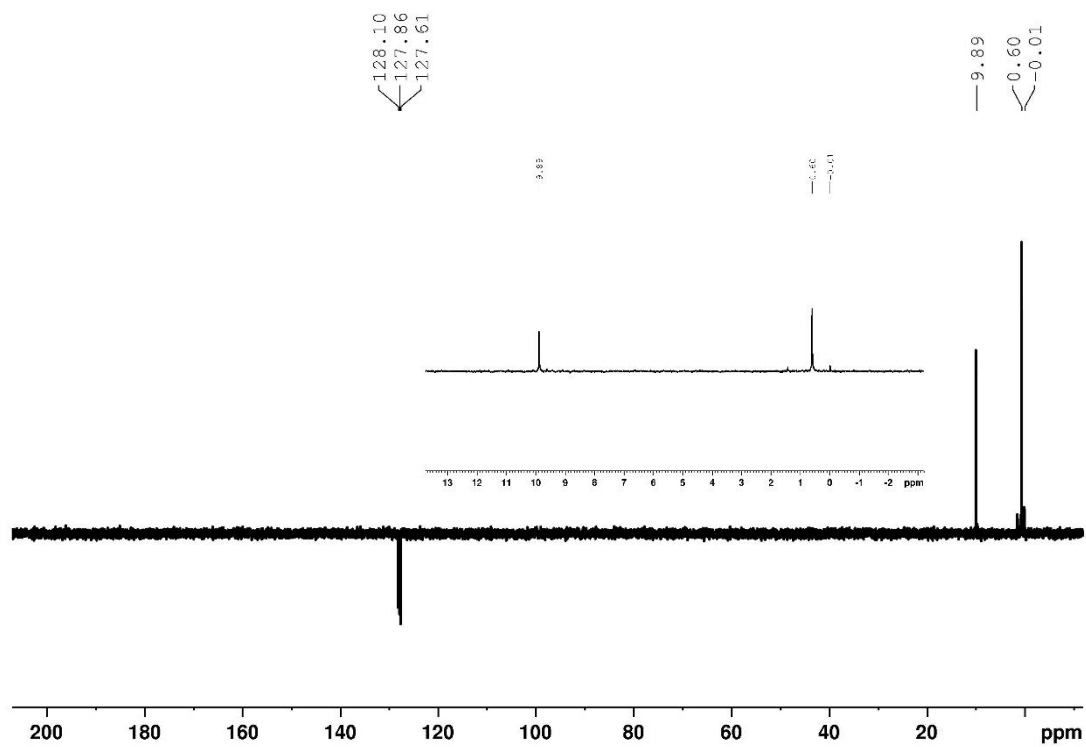


Figure S6 ^{13}C -NMR APT (100 MHz, 25 °C, C_6D_6) spectra of 4.

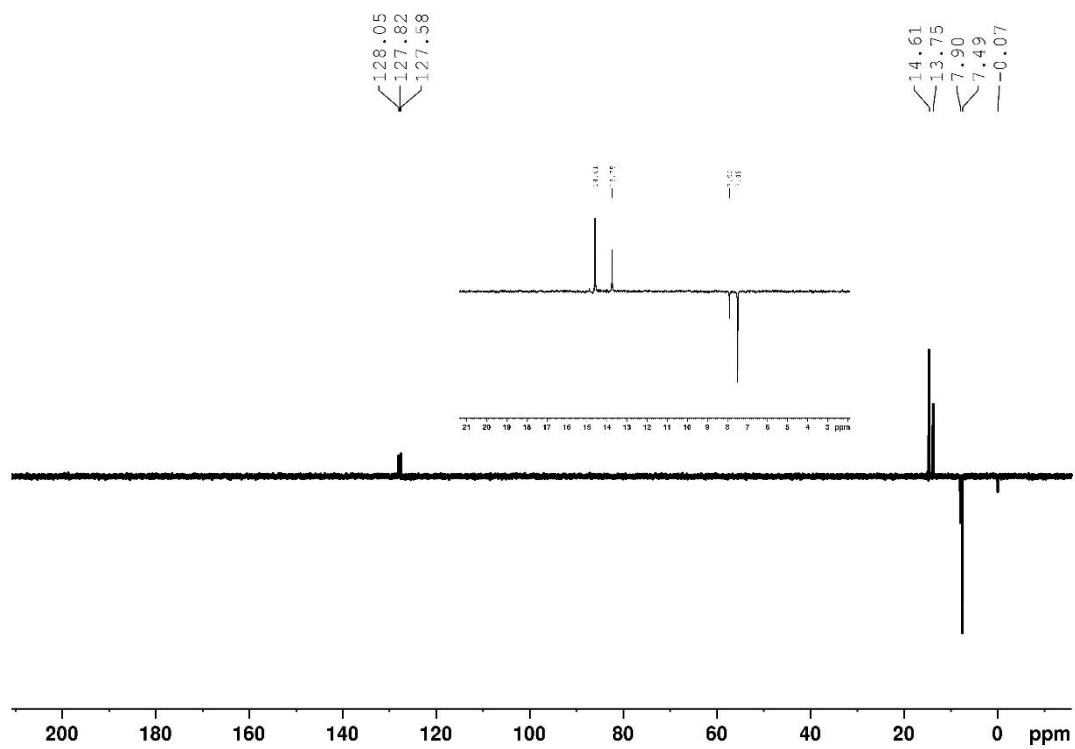


Figure S7 ^{13}C -NMR APT (100 MHz, 25 °C, C_6D_6) spectra of 5 (mixture).

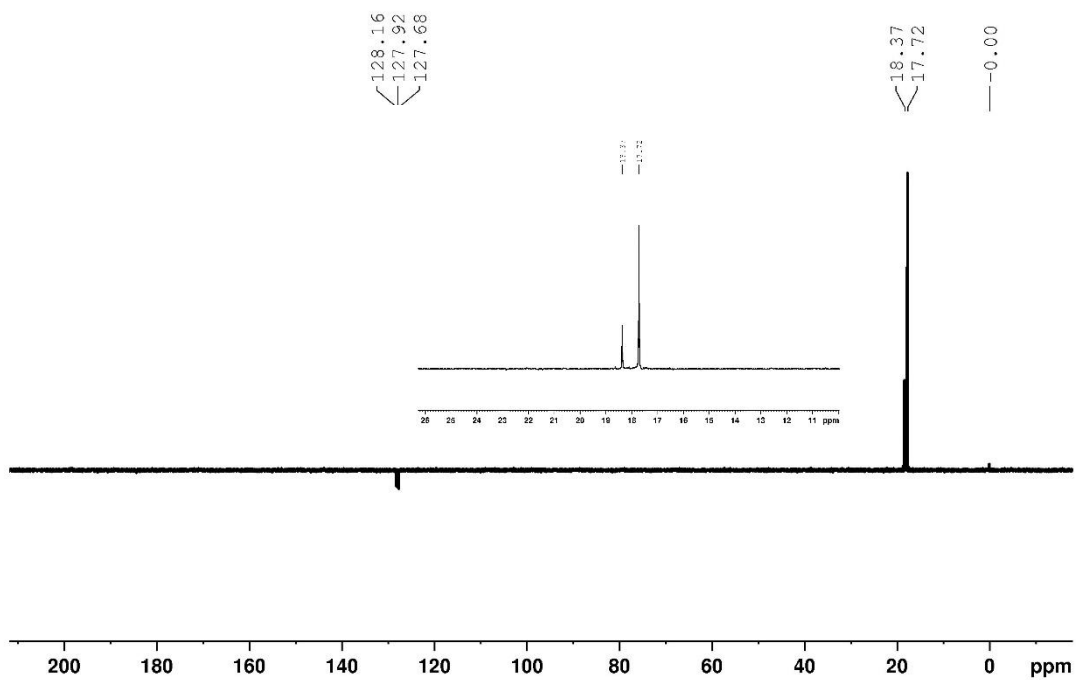


Figure S8 ^{13}C -NMR APT (100 MHz, 25 $^{\circ}\text{C}$, C_6D_6) spectra of **6**.

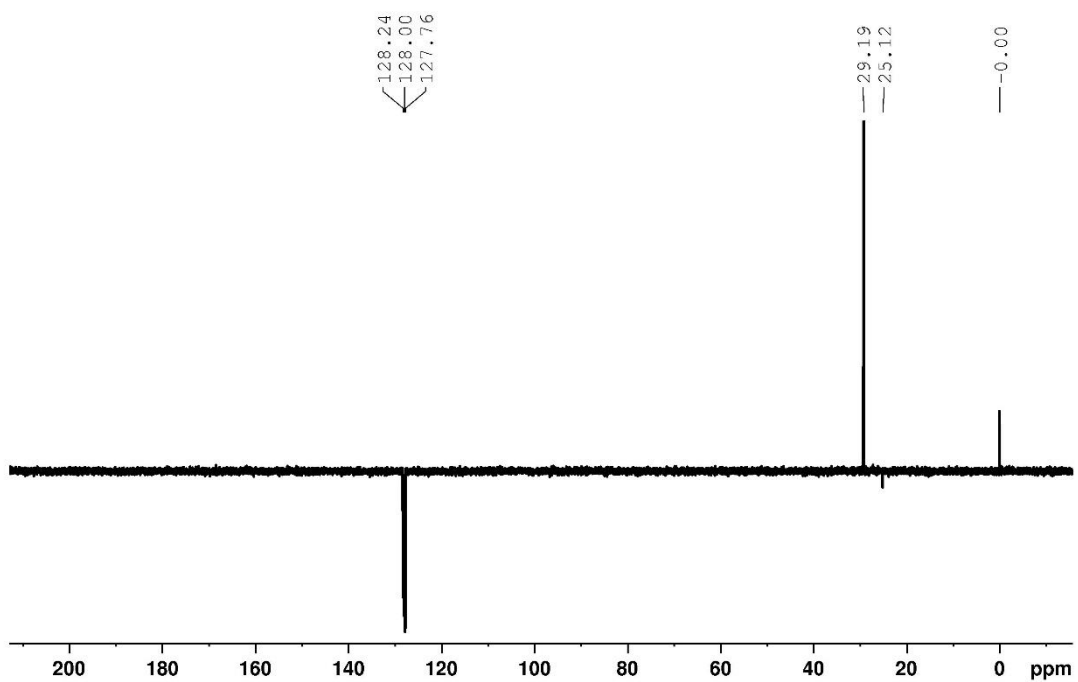


Figure S9 ^{13}C -NMR APT (100 MHz, 25 $^{\circ}\text{C}$, C_6D_6) spectra of **7**.

^1H -NMR (400 MHz, 25 $^{\circ}\text{C}$, C_6D_6) spectra of **7**.

2.3. ^{29}Si NMR spectra

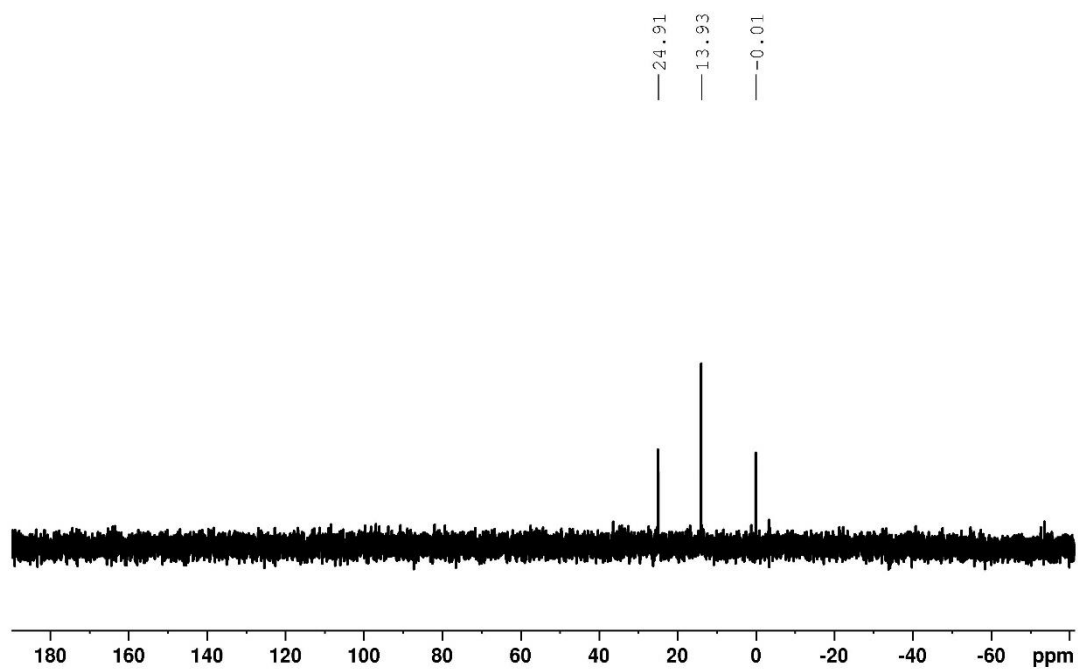


Figure S10 ^{29}Si -NMR (80 MHz, 25 °C, C_6D_6) spectra of **4**.

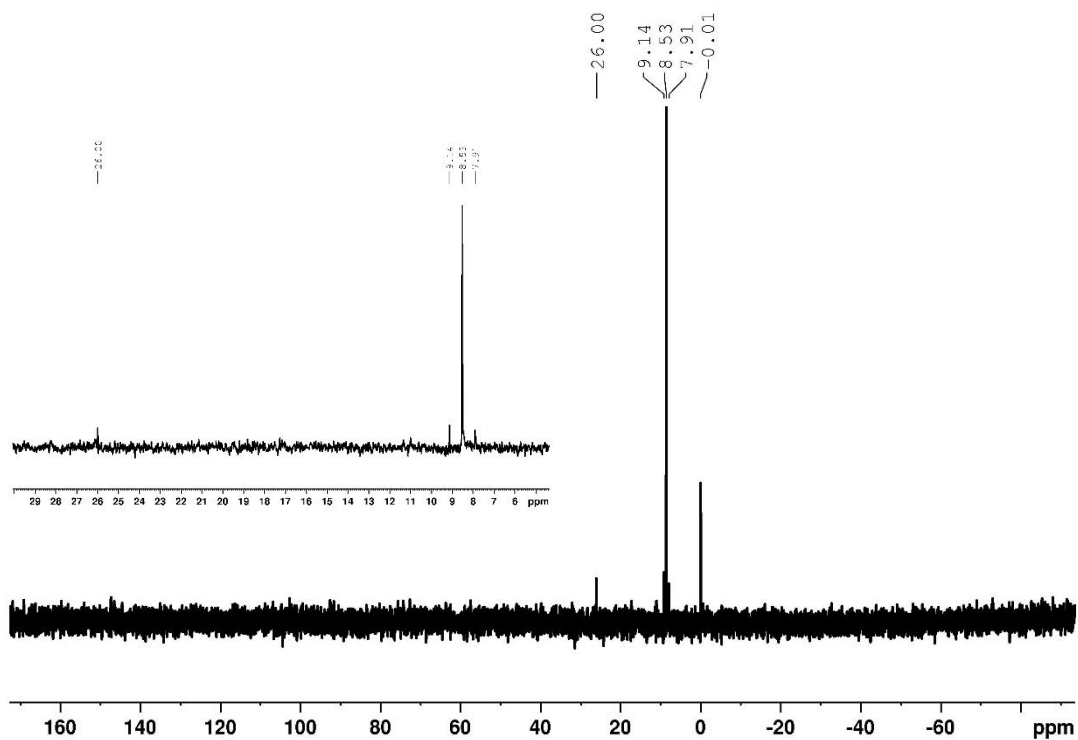


Figure S11 ^{29}Si -NMR (80 MHz, 25 °C, C_6D_6) spectra of **5** (mixture).

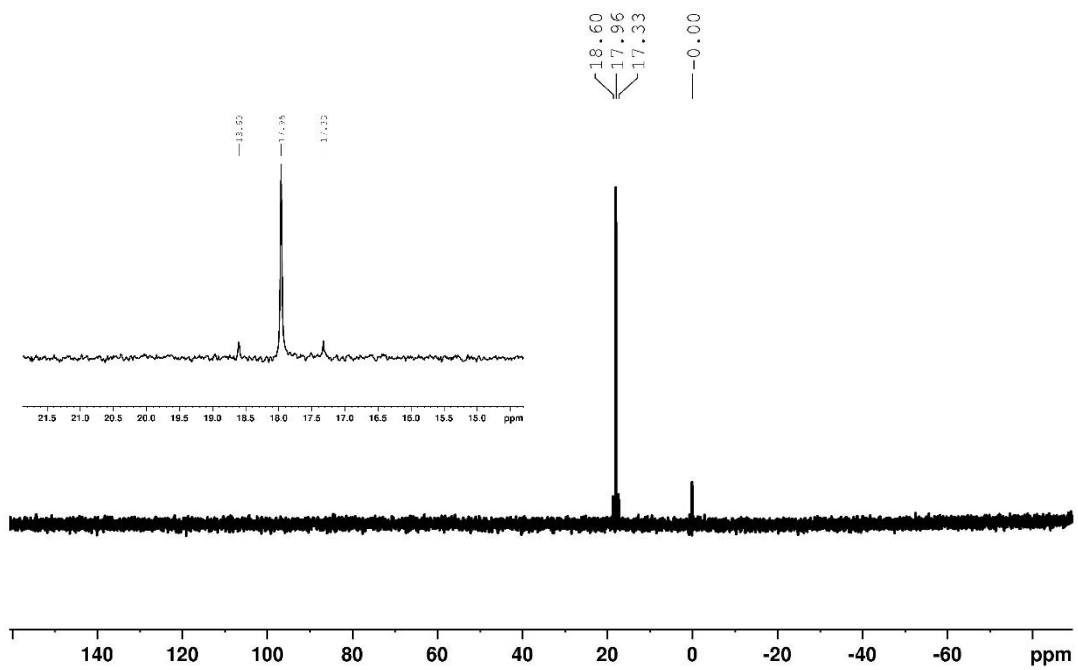


Figure S12 ^{29}Si -NMR (80 MHz, 25 °C, C_6D_6) spectra of **6**.

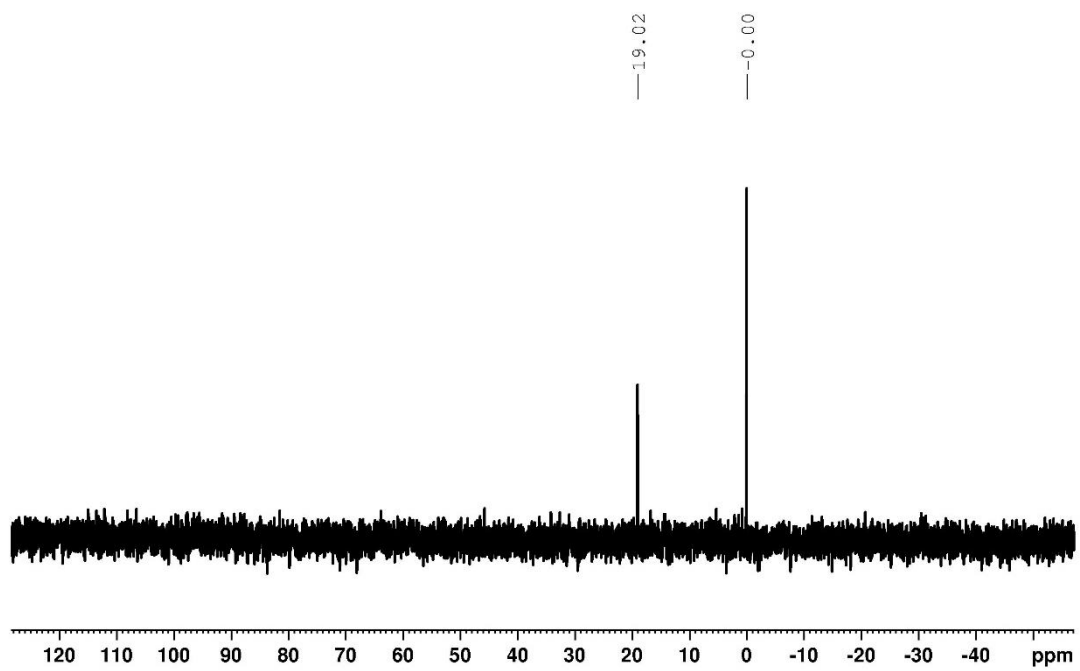


Figure S13 ^{29}Si -NMR (80 MHz, 25 °C, C_6D_6) spectra of **7**.

2.4. ^{77}Se NMR spectra

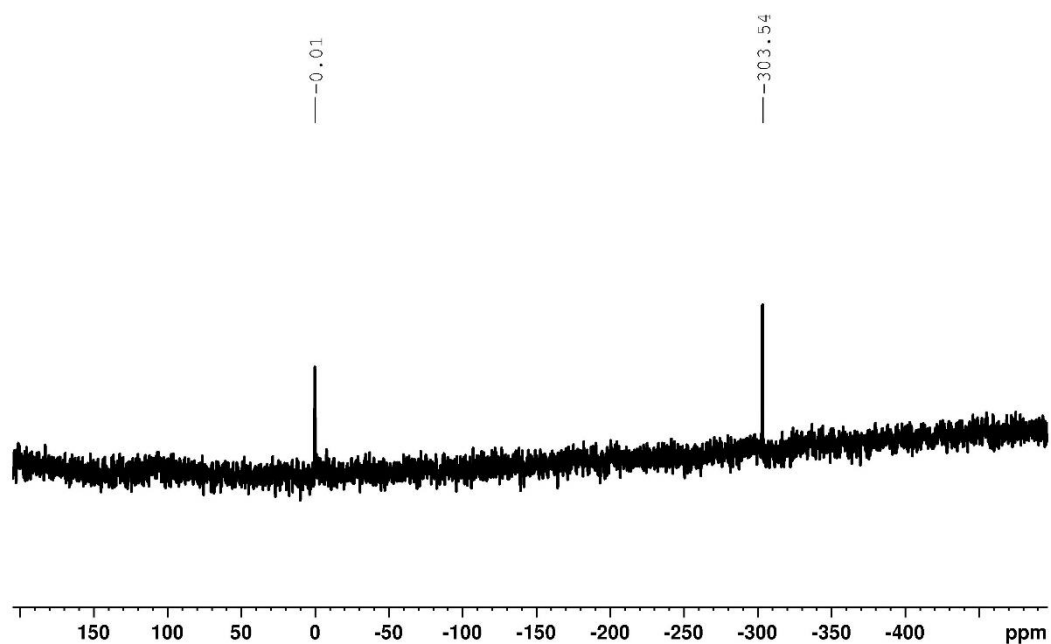


Figure S14 ^{77}Se -NMR (76 MHz, 25 °C, C_6D_6) spectra of 4.

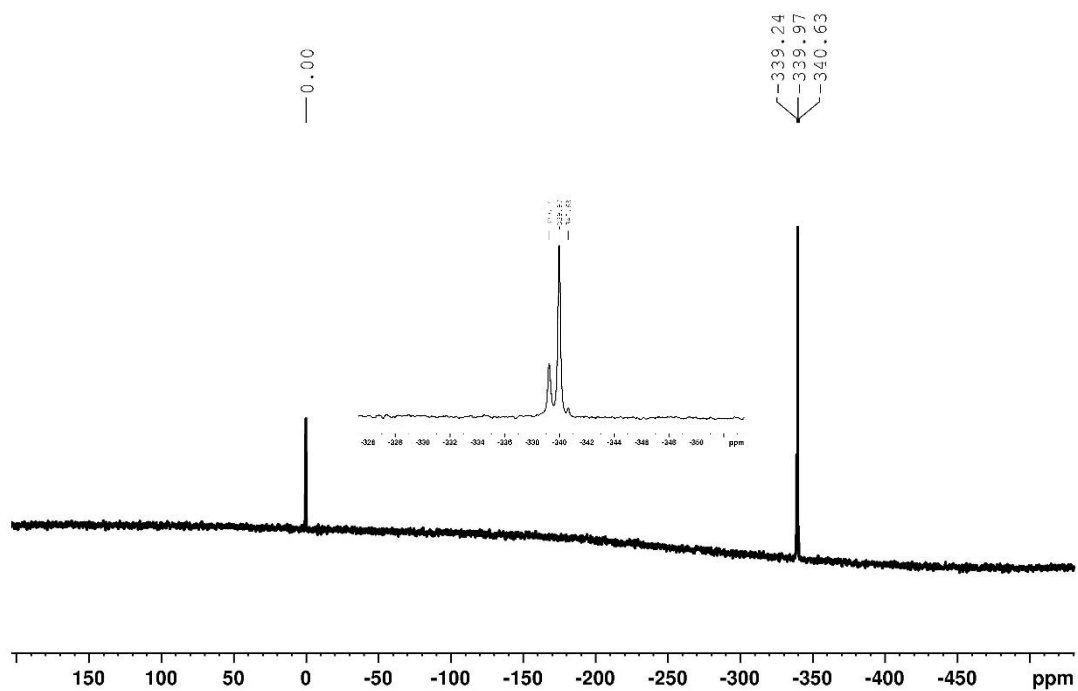


Figure S15 ^{77}Se -NMR (76 MHz, 25 °C, C_6D_6) spectra of 5 (mixture).

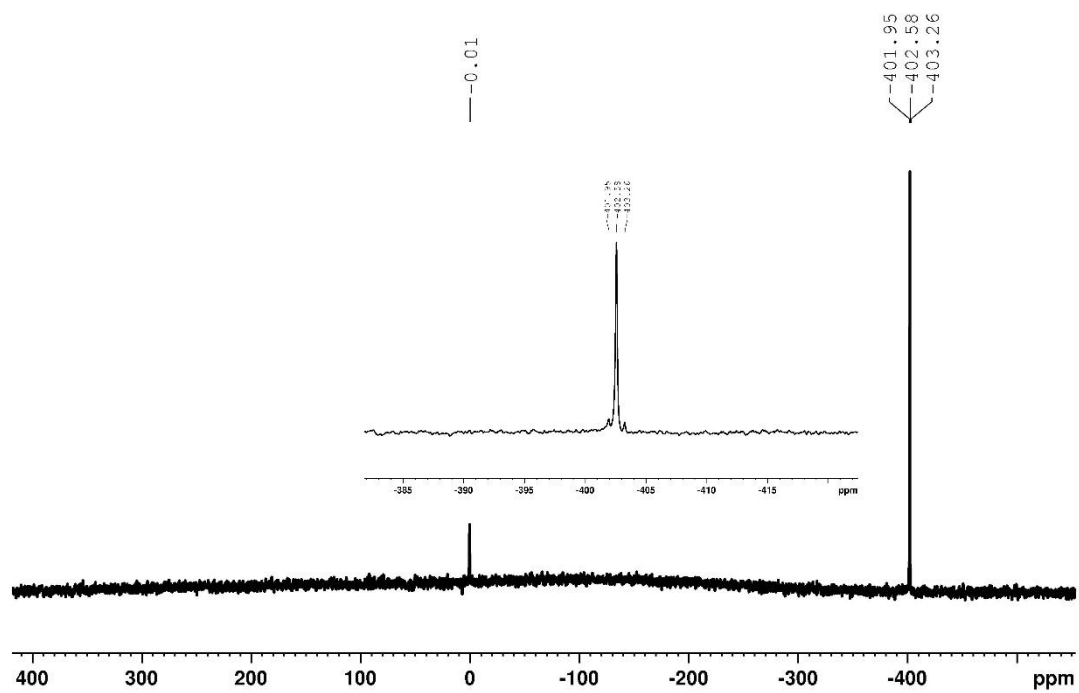


Figure S16 ^{77}Se -NMR (76 MHz, 25 °C, C_6D_6) spectra of 6.

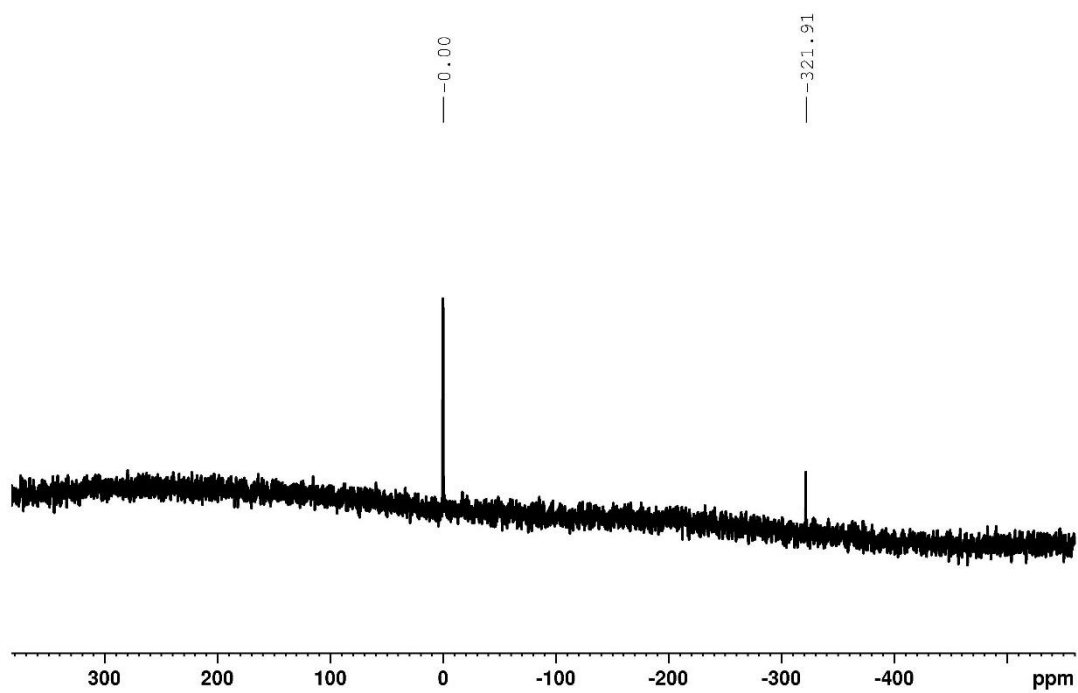


Figure S17 ^{77}Se -NMR (76 MHz, 25 °C, C_6D_6) spectra of 7.

3. GC/MS records

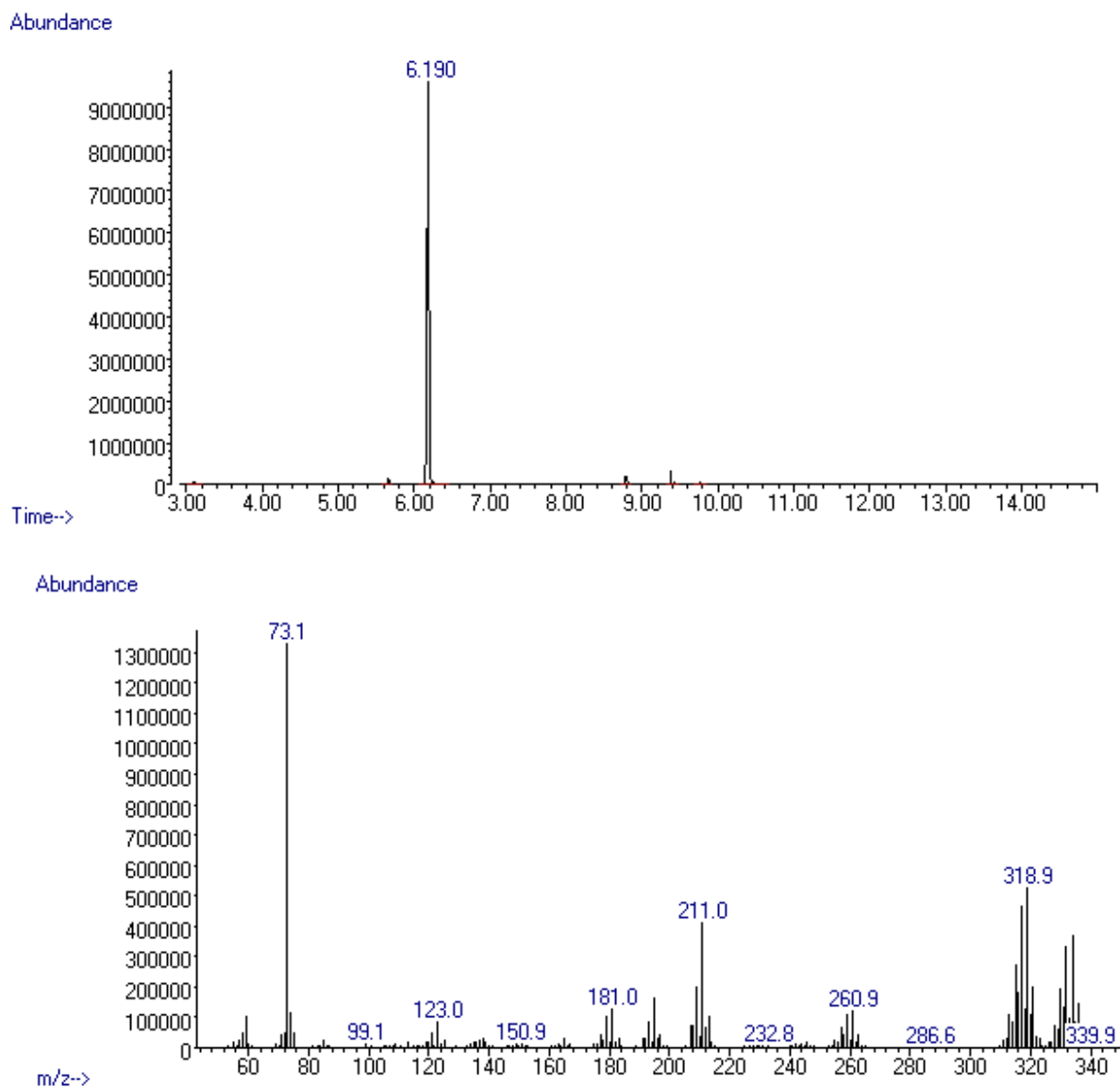


Figure S18 GC/MS record of 4.

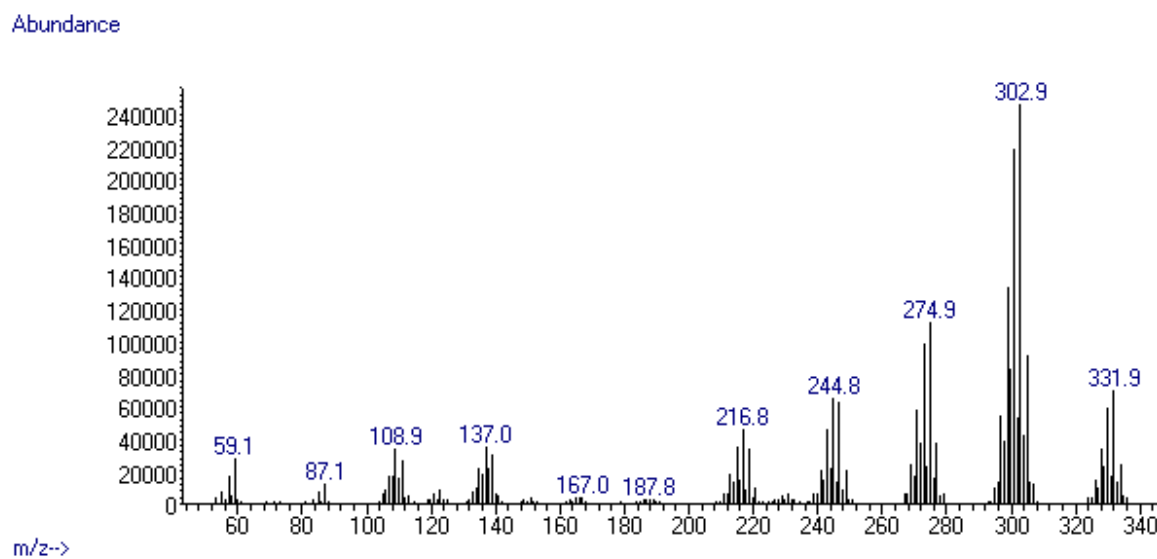
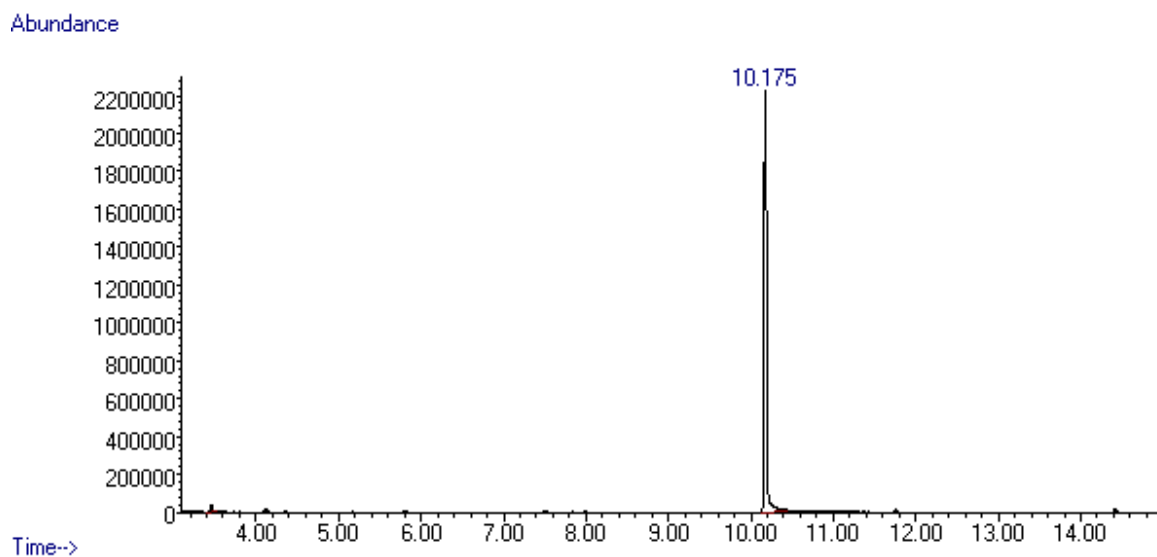


Figure S19 GC/MS record of 5 (mixture).

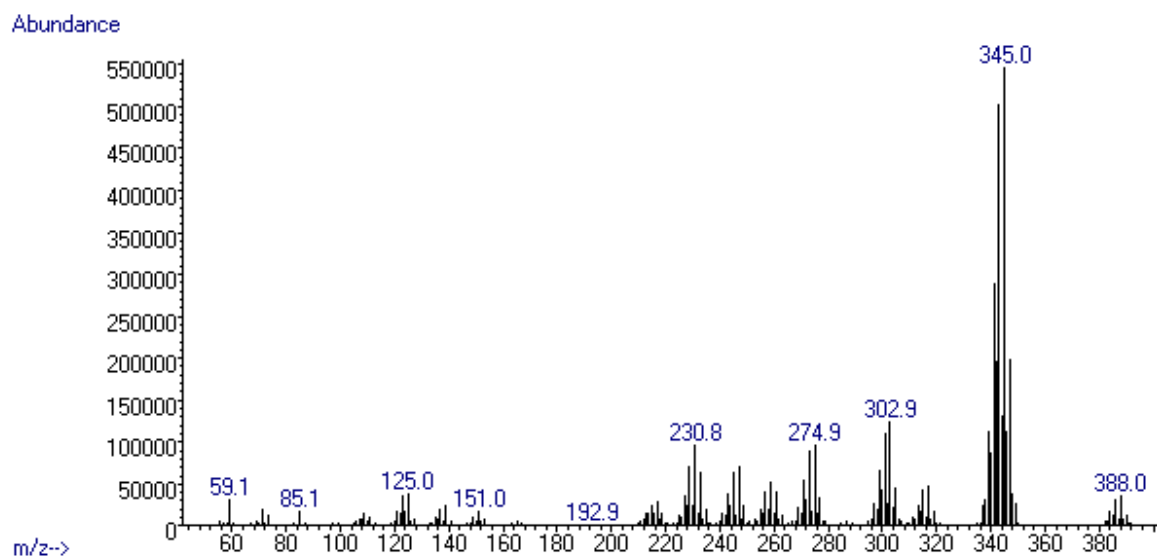
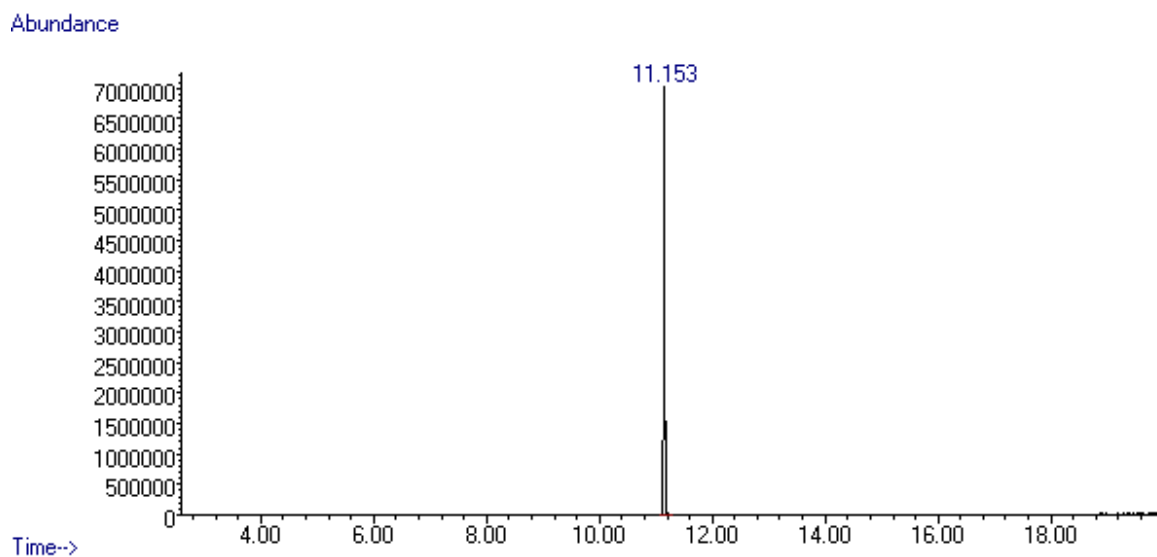


Figure S20 GC/MS record of 6.

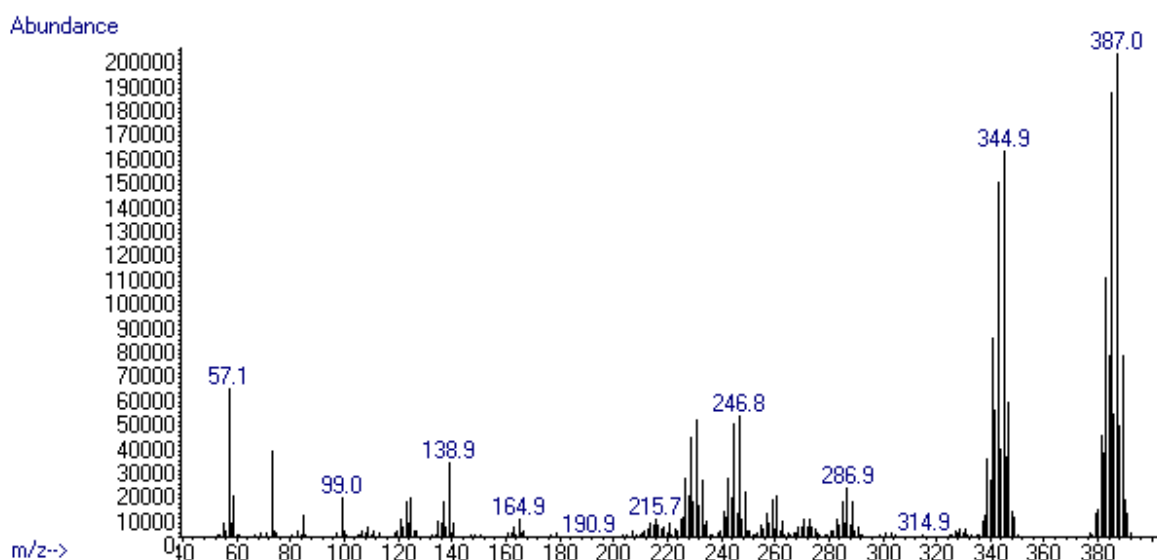
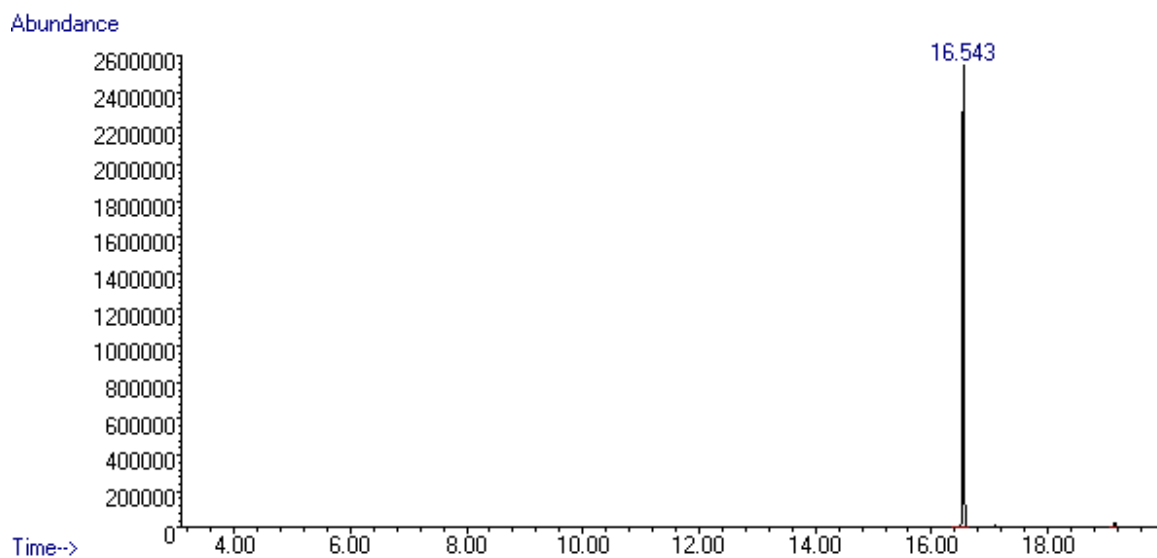


Figure S21 GC/MS record of 7.

4. DSC thermograms

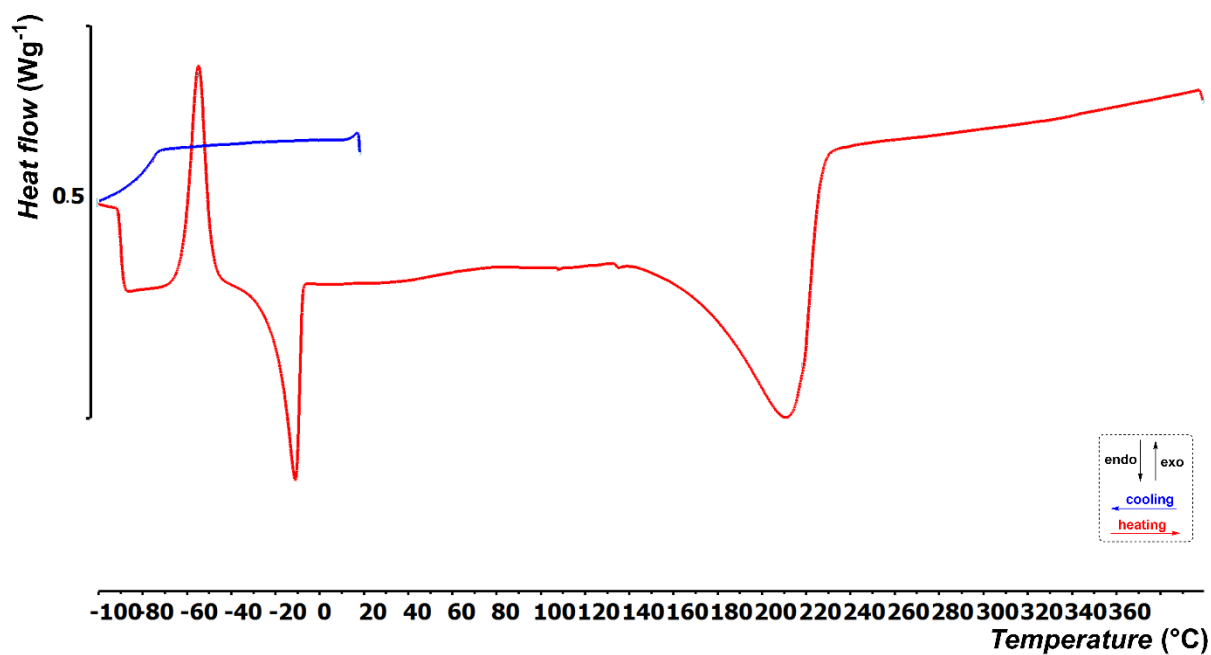


Figure S22 DSC curve of 4.

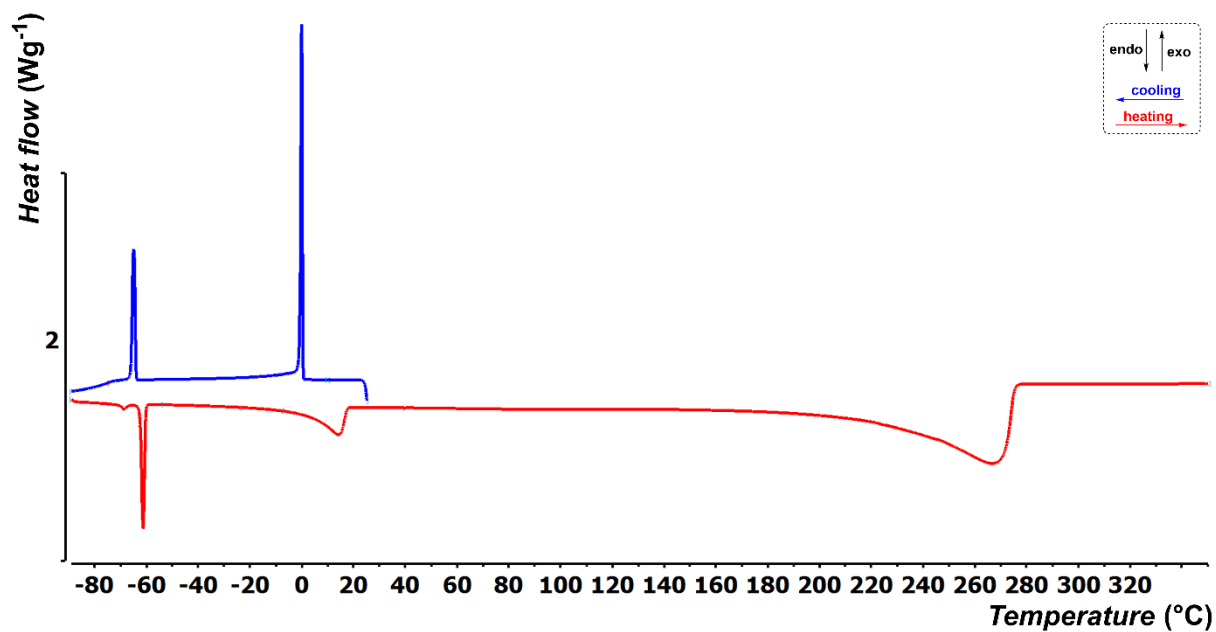


Figure S23 DSC curve of 5 (mixture)

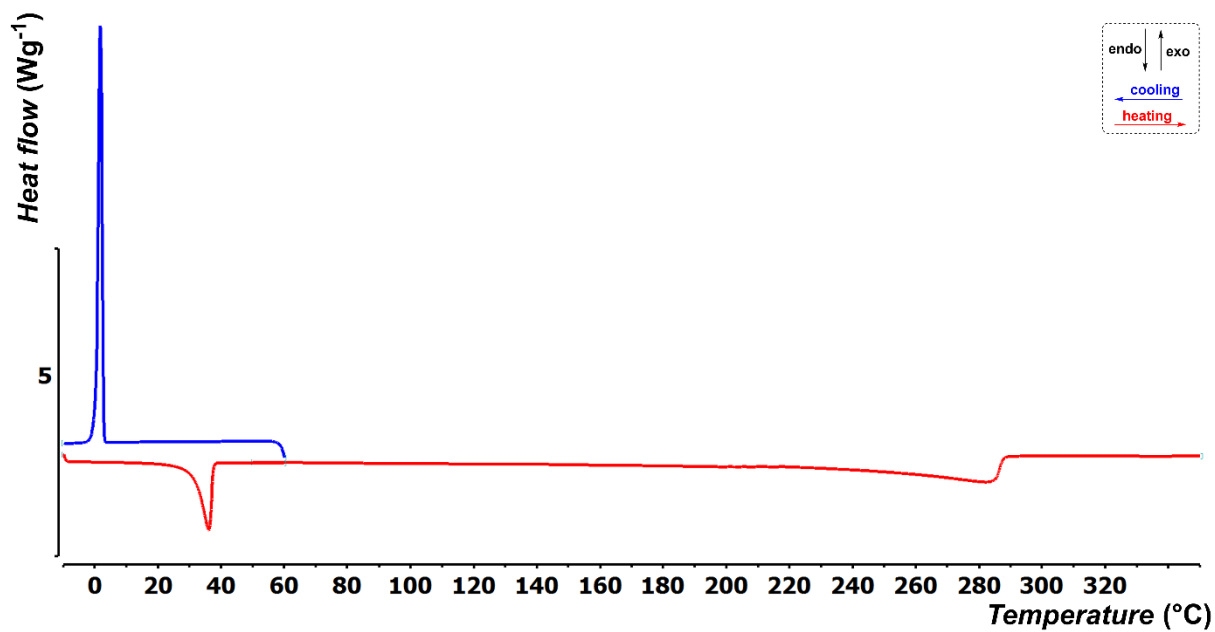


Figure S24 DSC curve of 6.

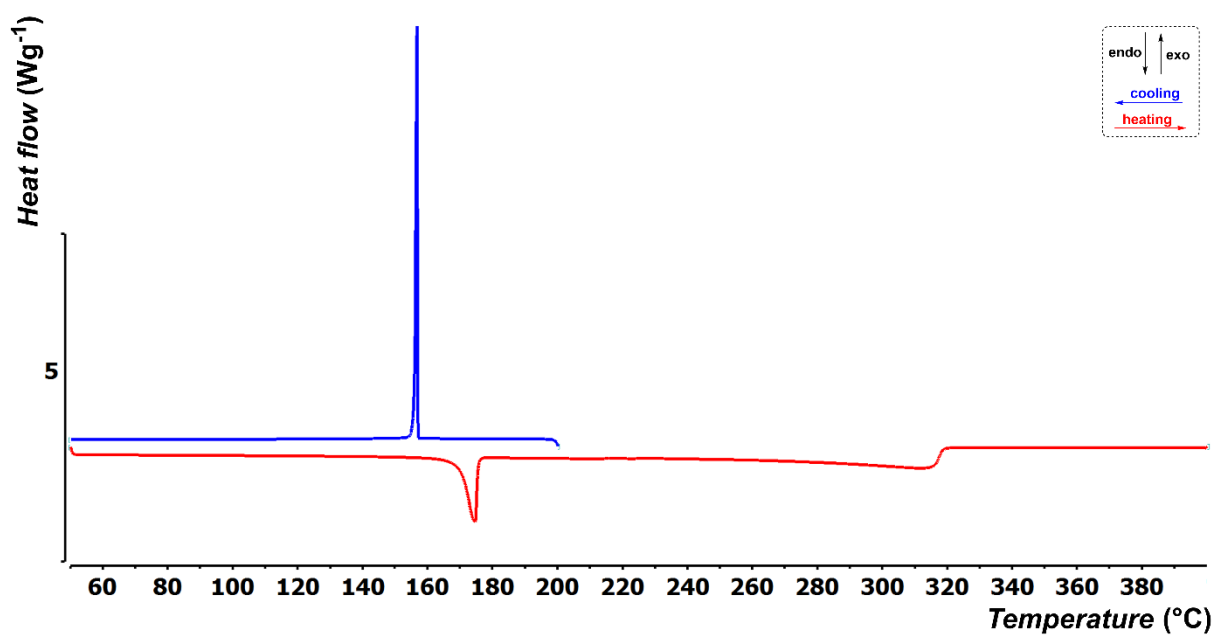


Figure S25 DSC curve of 7.

5. TGA

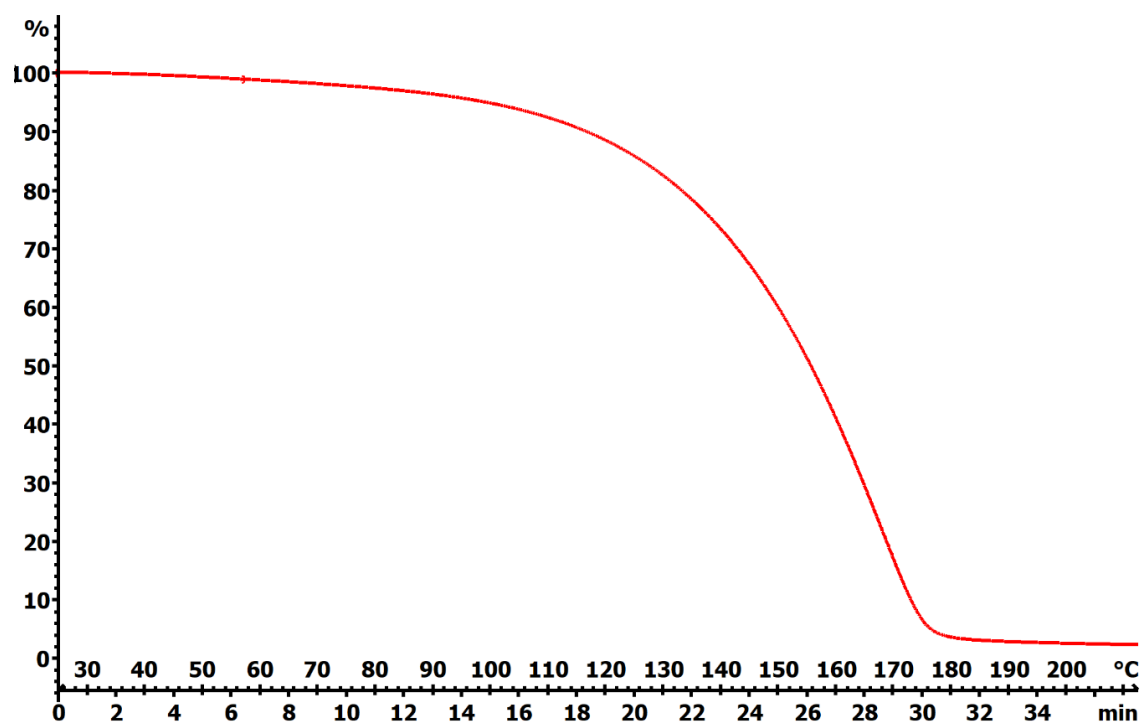


Figure S26 TGA curve of 4.

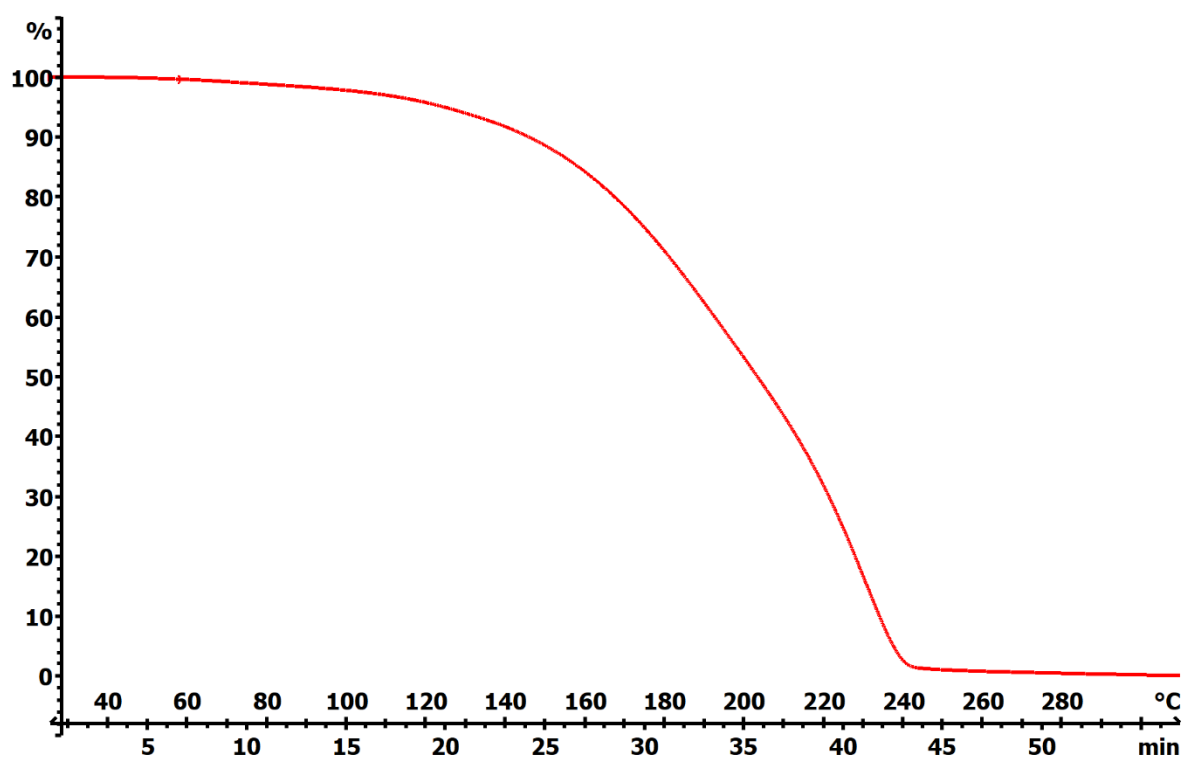


Figure S27 TGA curve of 5 (mixture).

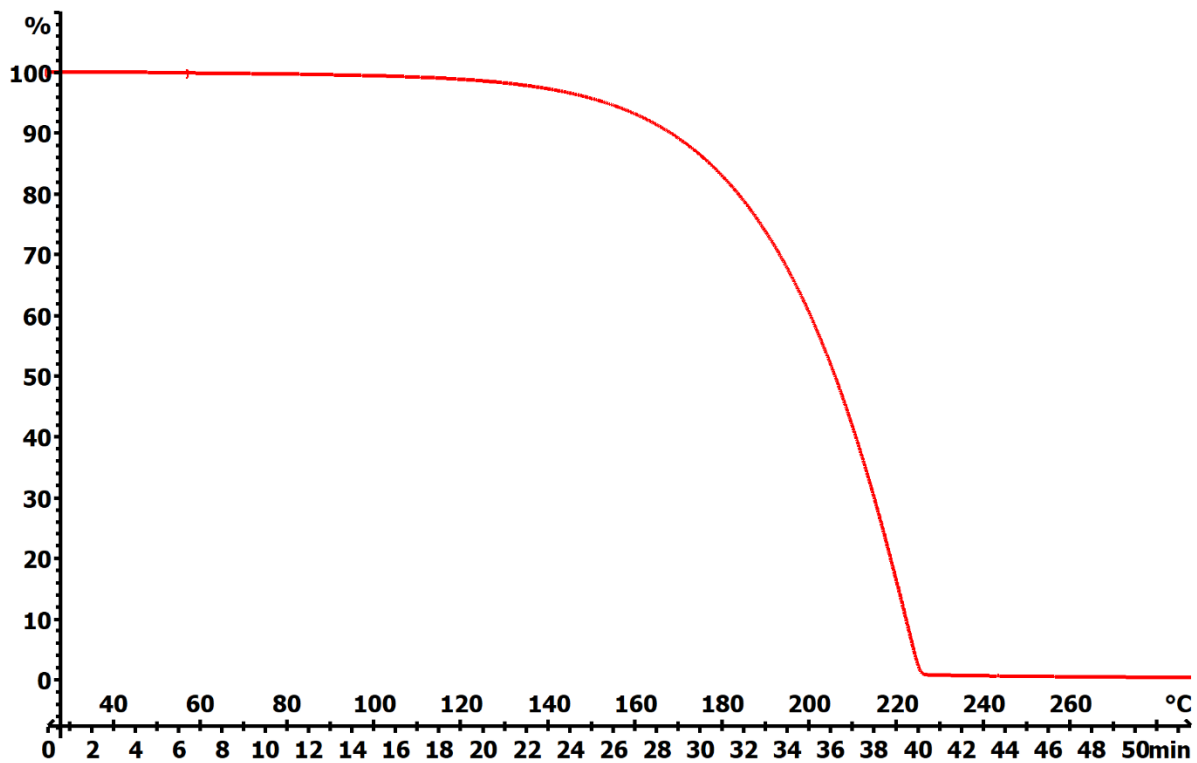


Figure S28 TGA curve of 6.

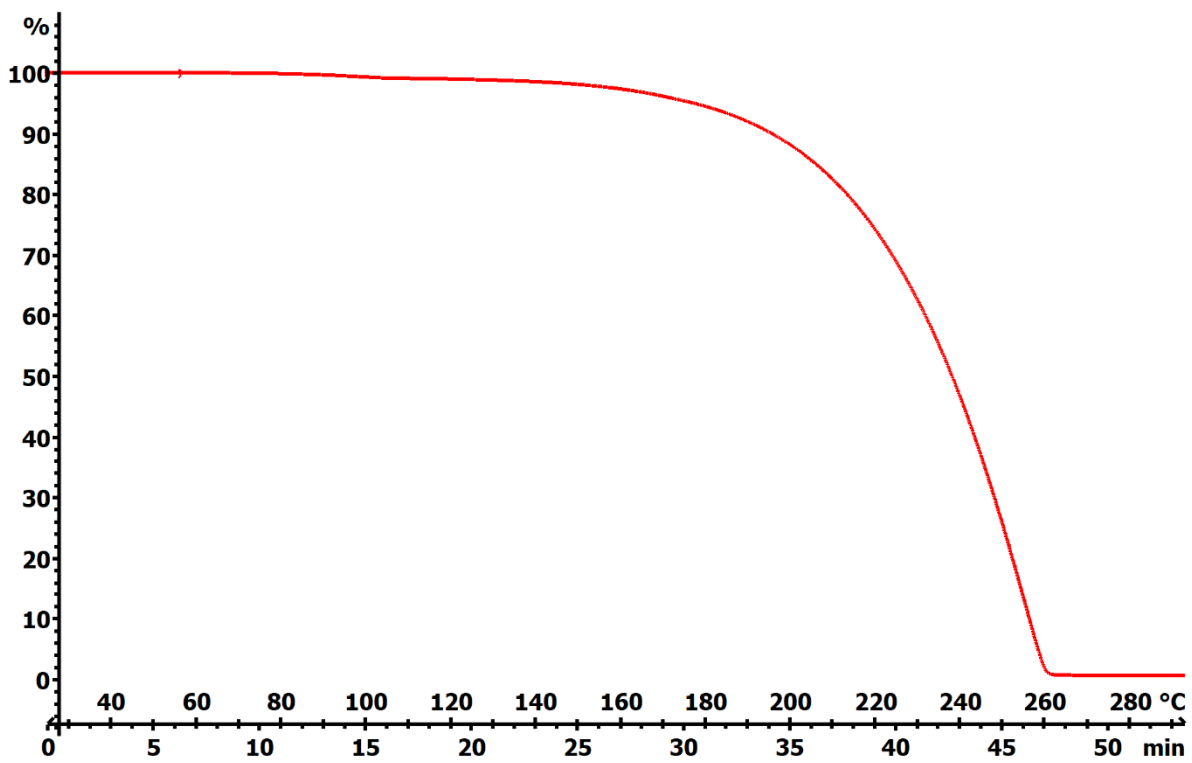


Figure S29 TGA curve of 7.

6. SEM

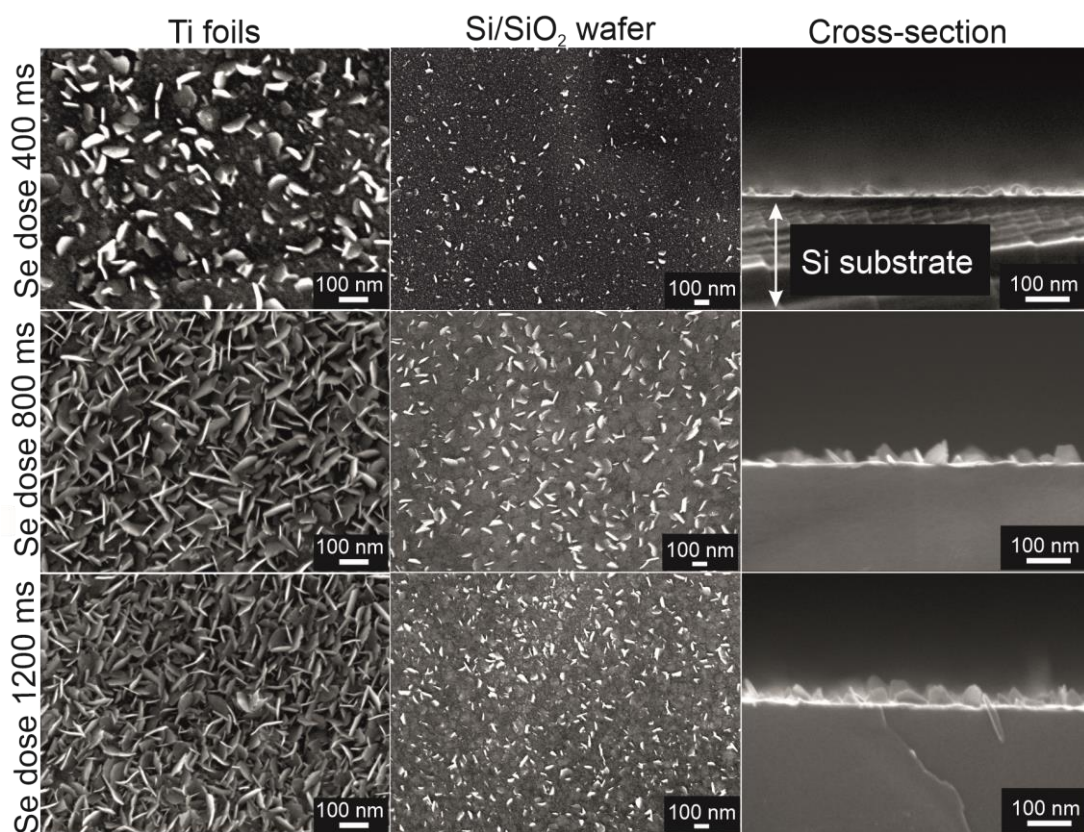


Figure S30 Left and central column: SEM top view images of as deposited MoSe₂ at 300°C using Se precursor **6** upon 800 ALD cycles on annealed titanium foil and silicon wafer, respectively, applying constant Mo dose (800 ms) and different Se dosing, namely, 400, 800 and 1200 ms; Right column: the cross-sectional SEM images of the Si wafers corresponding to samples from the central column.

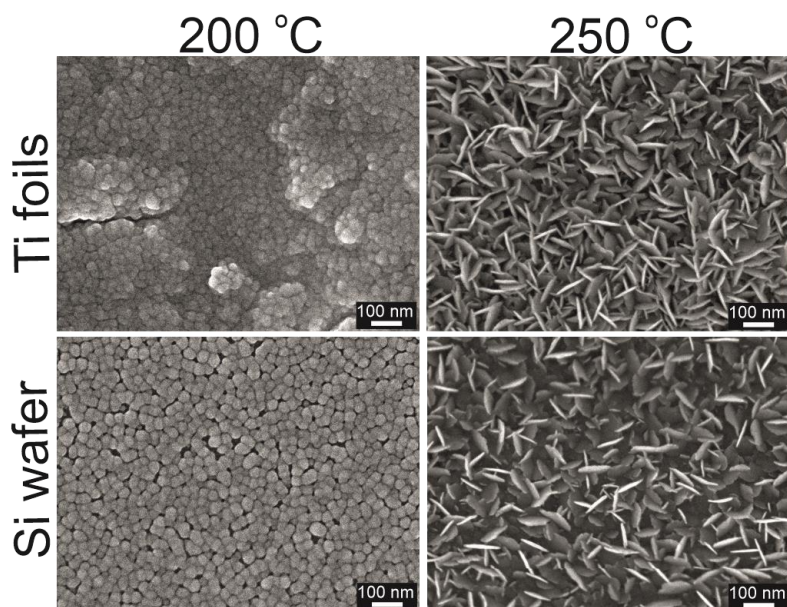


Figure S31 SEM top view images of MoSe₂ deposited at 200 and 250°C using Se precursor **6** on annealed titanium foil and silicon wafer (800 ms Se dose) upon 800 ALD cycles.

7. Raman spectroscopy

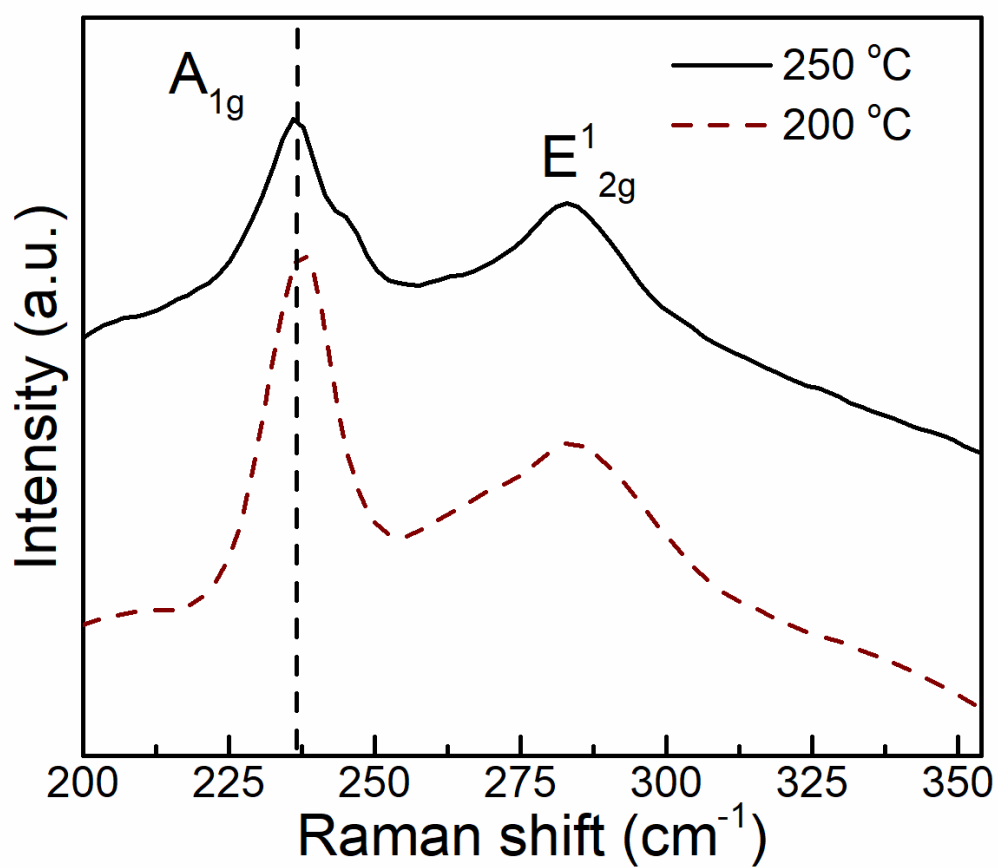


Figure S32 Raman spectra obtained from MoSe₂ deposited at 200 and 250 °C using Se precursor 6 upon 800 ALD cycles on annealed titanium foil (800 ms Se dose).

8. XPS

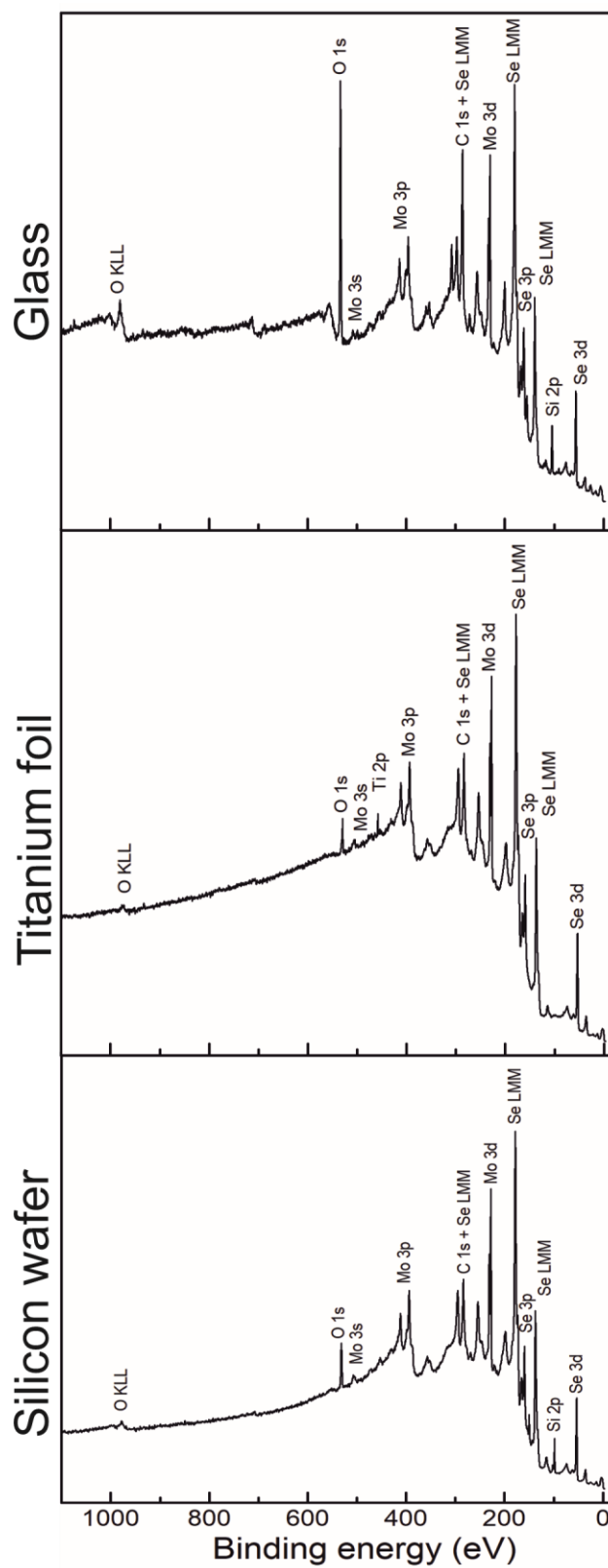


Figure S33 XPS survey spectra of ALD MoSe₂ deposited at 300 °C using Se precursor **6** upon 800 ALD cycles (800 ms Se dose) on different substrates: glass, titanium foil and silicon wafer.

