Supplementary Materials

Regioselective One-Pot Synthesis of Novel Functionalized Organoselenium Compound By Bis-Alkoxyselenenylation of Alkenes with Selenium Dibromide and Alcohols

Vladimir A. Potapov, Maxim V. Musalov, Alfiya G. Khabibulina, Arkady A. Maylyan, Tatyana N. Borodina, Sergey V. Zinchenko and Svetlana V. Amosova*

A. E. Favorsky Irkutsk Institute of Chemistry, Siberian Division of The Russian Academy of Sciences, 1 Favorsky Str., Irkutsk 664033, Russia

*Correspondence: amosova@irioch.irk.ru

Table of Contents

General Information

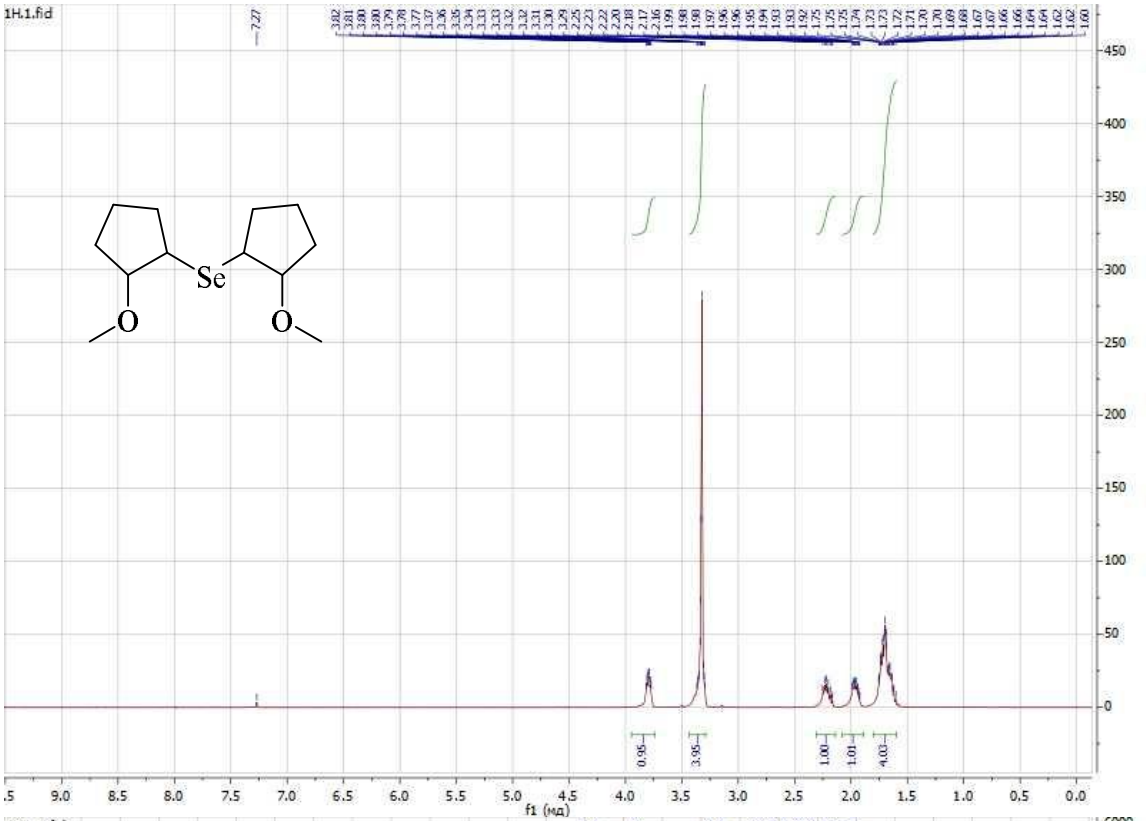
X-ray diffraction experiments were carried out on a Bruker D8 Venture Photon 100 CMOS diffractometer with Mo-K_α radiation (λ = 0.71073 Å). X-Ray crystallographic data for compound 2 (CCDC 2207651) are shown in Supplementary Info. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html. ^{1}H (400.1 MHz) and ^{13}C (100.6 MHz) NMR spectra were recorded on a Bruker DPX-400 spectrometer (Bruker BioSpin GmbH, Rheinstetten, Germany) in 5-10% solution in $CDCl_3$. ^{1}H and ^{13}C chemical shifts (δ) are reported in parts per million (ppm), relative to the residual solvent peak of $CDCl_3$ (δ = 7.27 and 77.16 ppm in ^{1}H and ^{13}C-NMR, respectively).

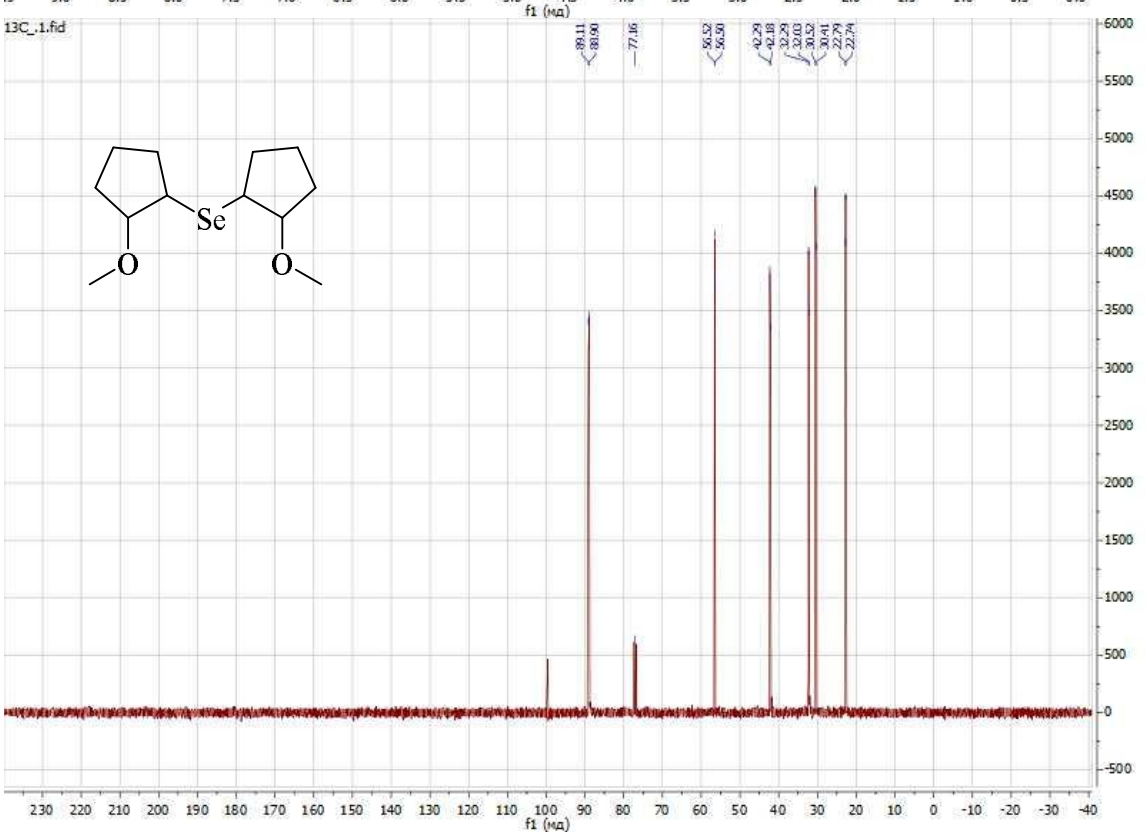
Elemental analysis was performed on a Thermo Scientific Flash 2000 Elemental Analyzer.

The organic solvents were dried and distilled according to standard procedures.

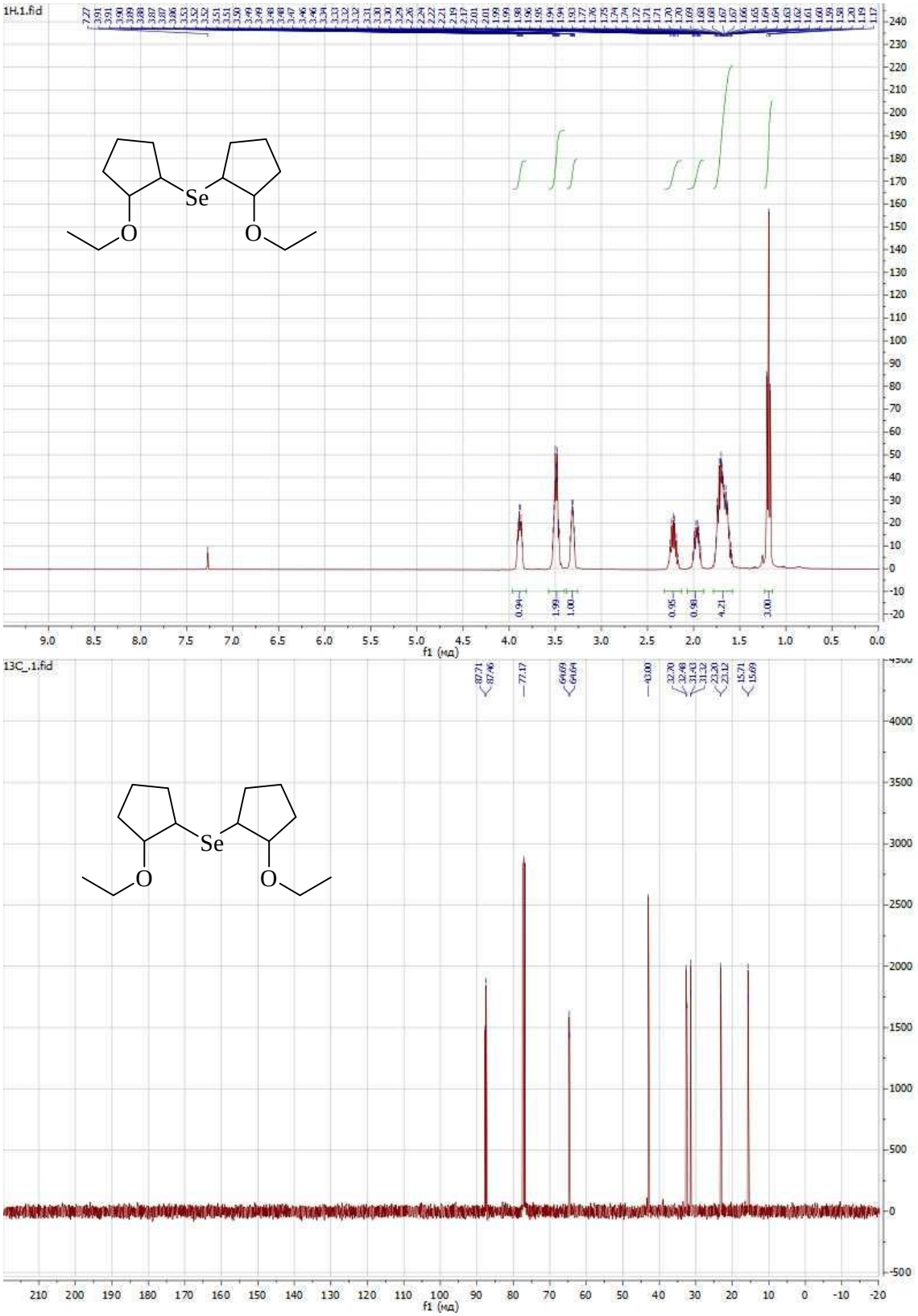
Examples of ^{1}H and ^{13}C-NMR Spectra

^{1}H- and ^{13}C-NMR spectra of compound 3

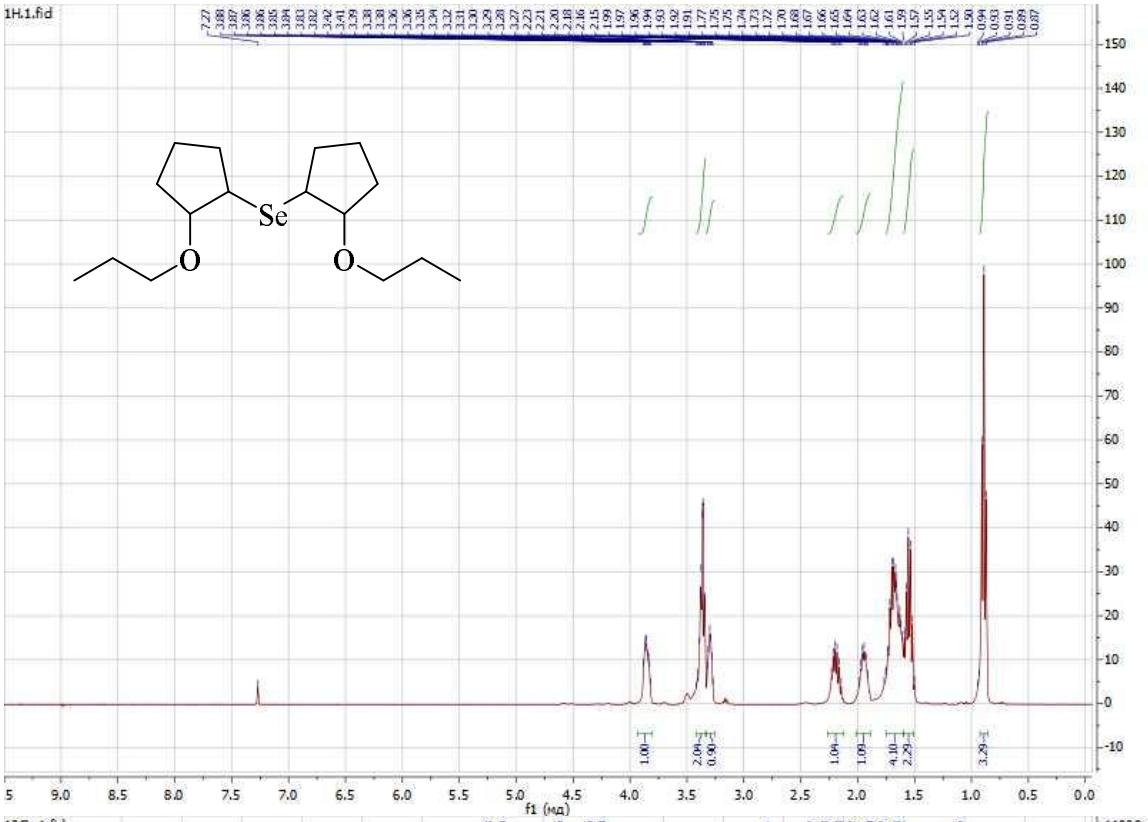


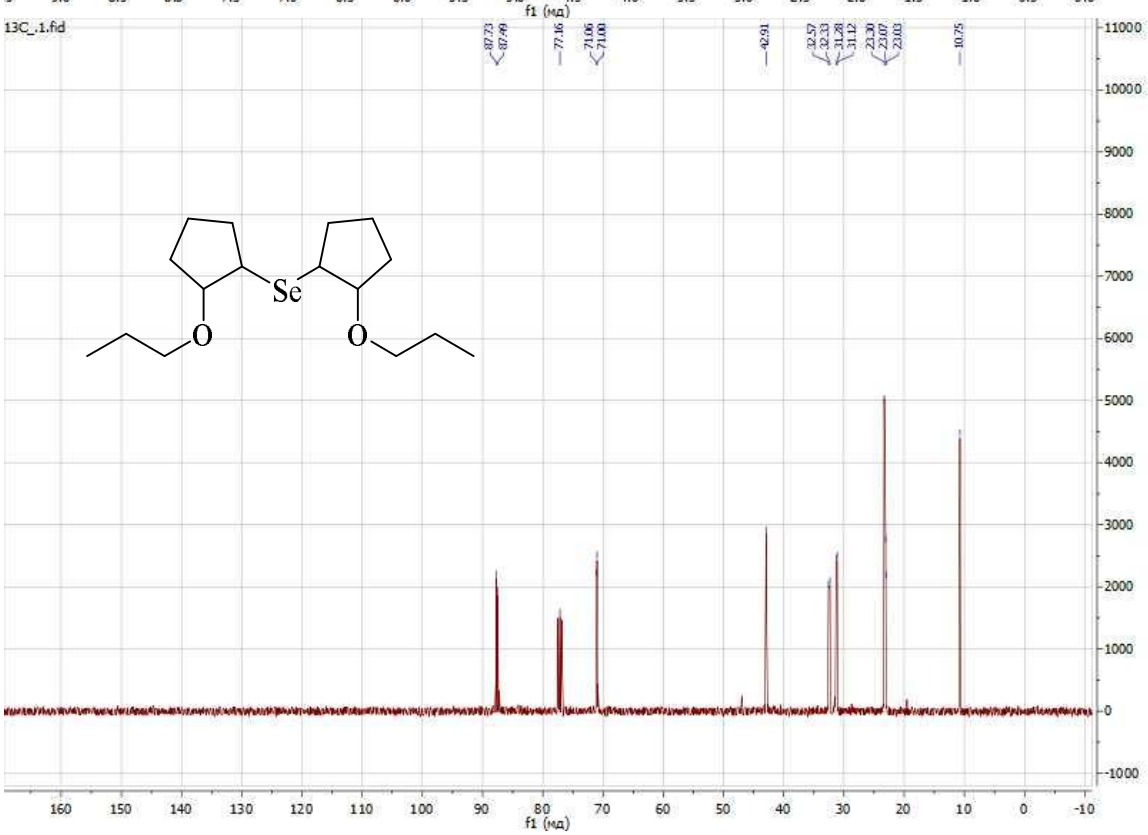


^{1}H- and ^{13}C-NMR spectra of compound 4

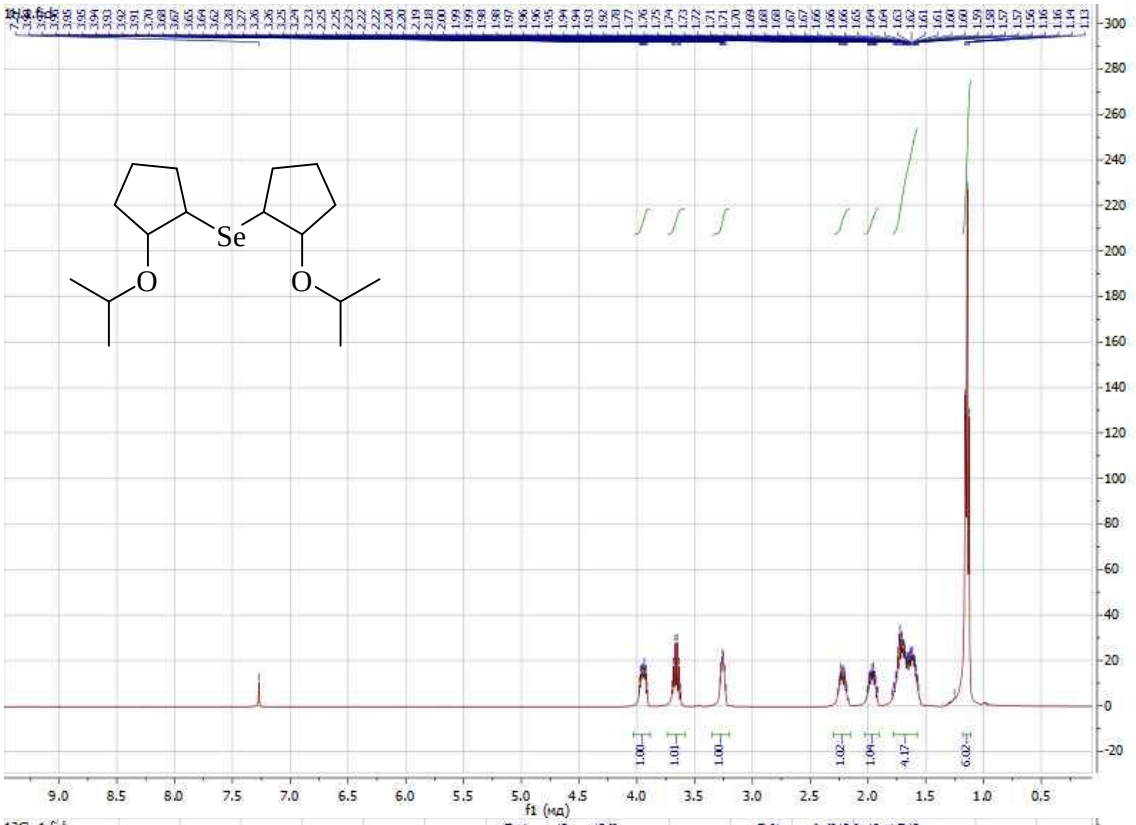


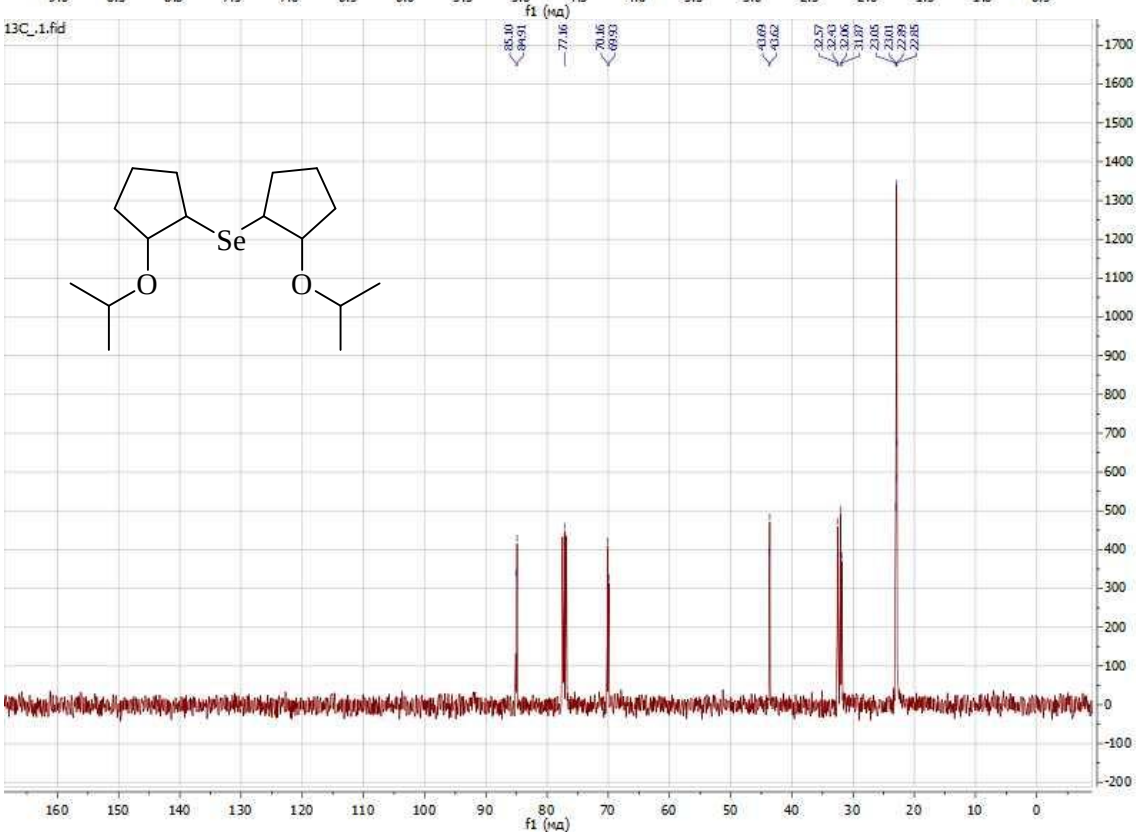
^{1}H- and ^{13}C-NMR spectra of compound 5



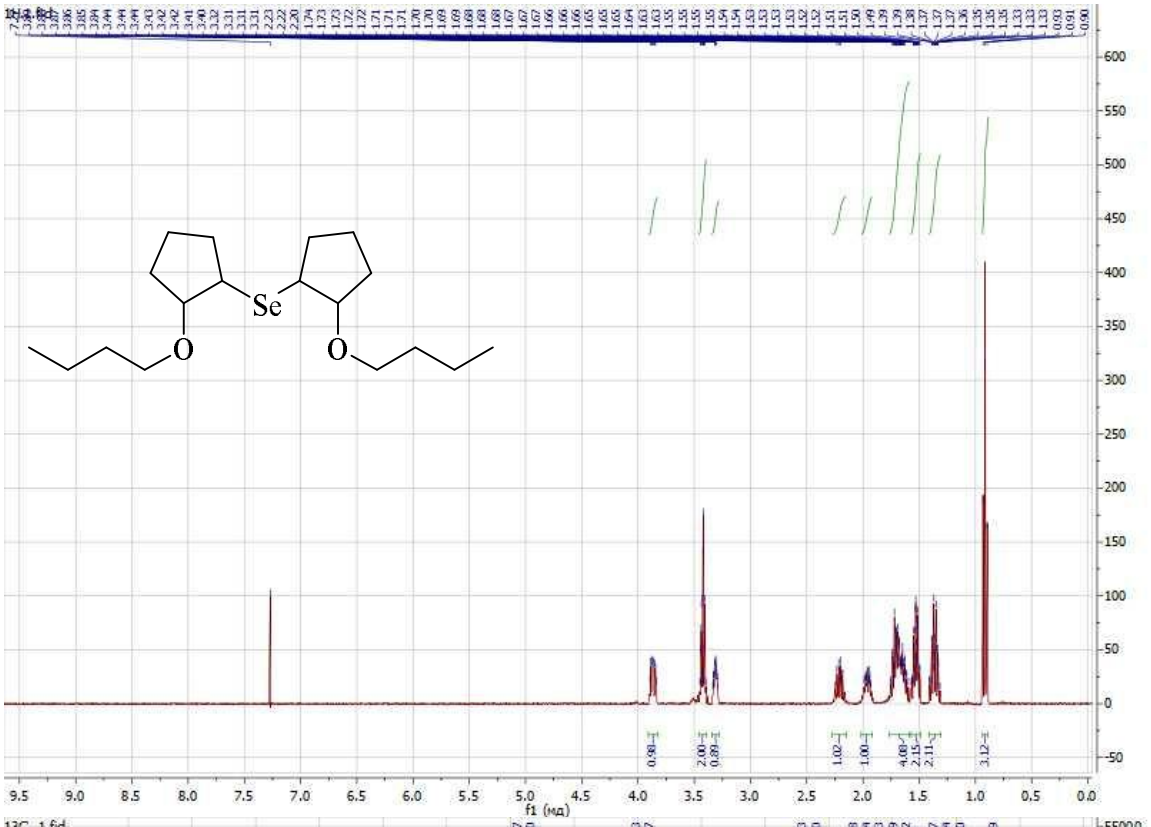


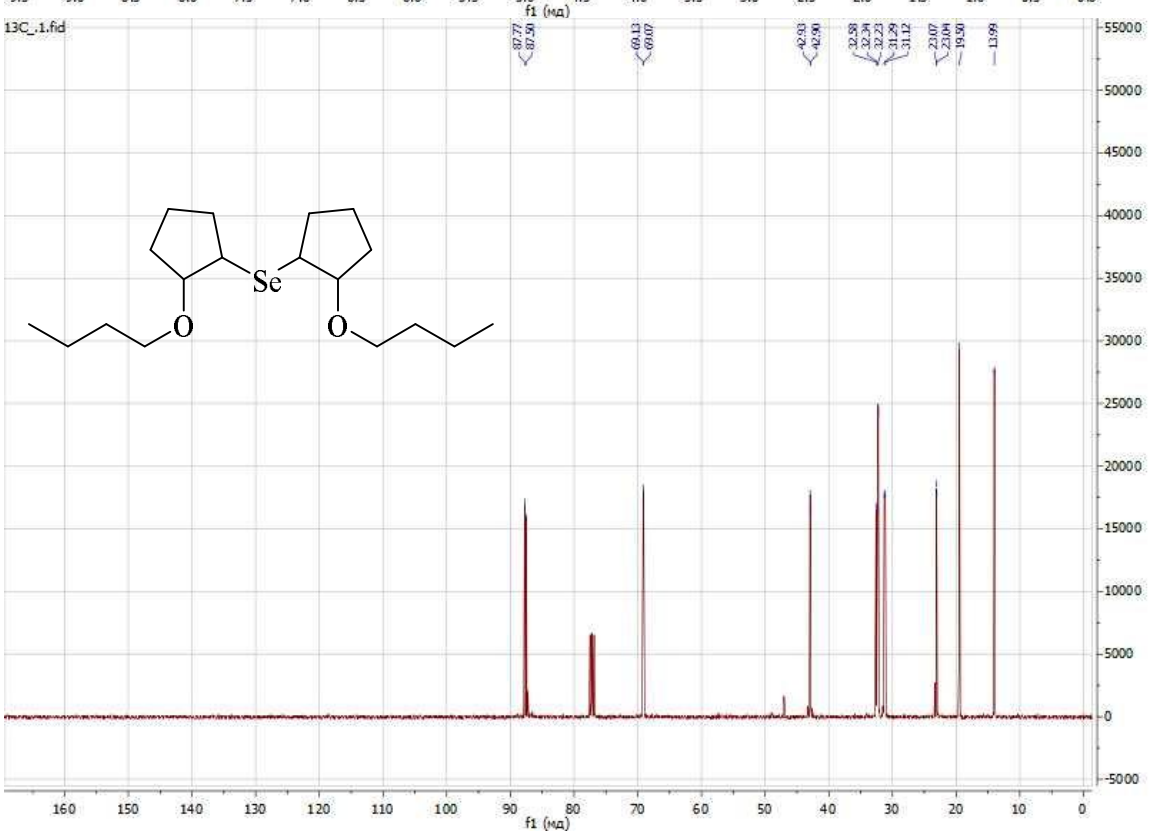
^{1}H- and ^{13}C-NMR spectra of compound 6



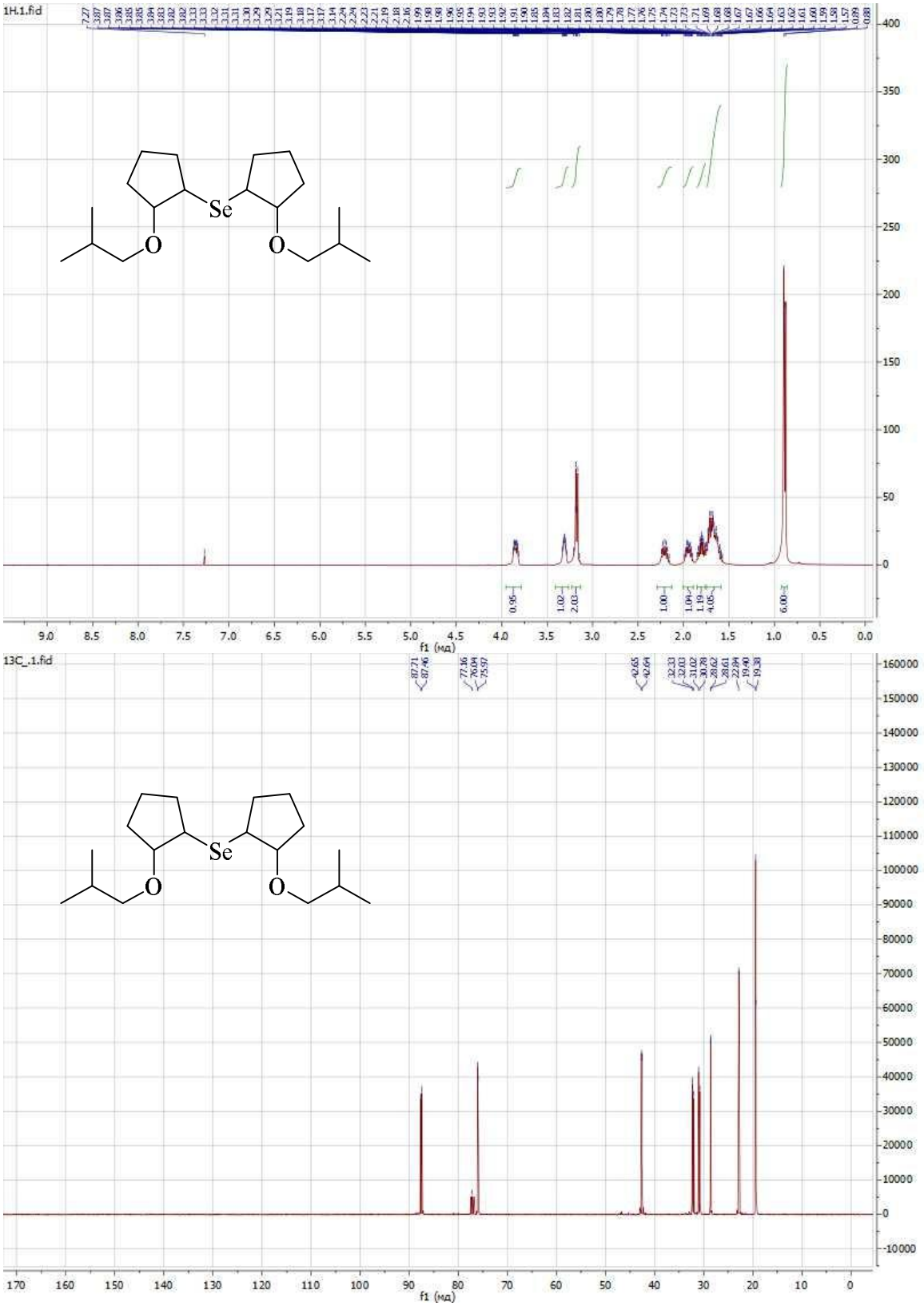


^{1}H- and ^{13}C-NMR spectra of compound 7

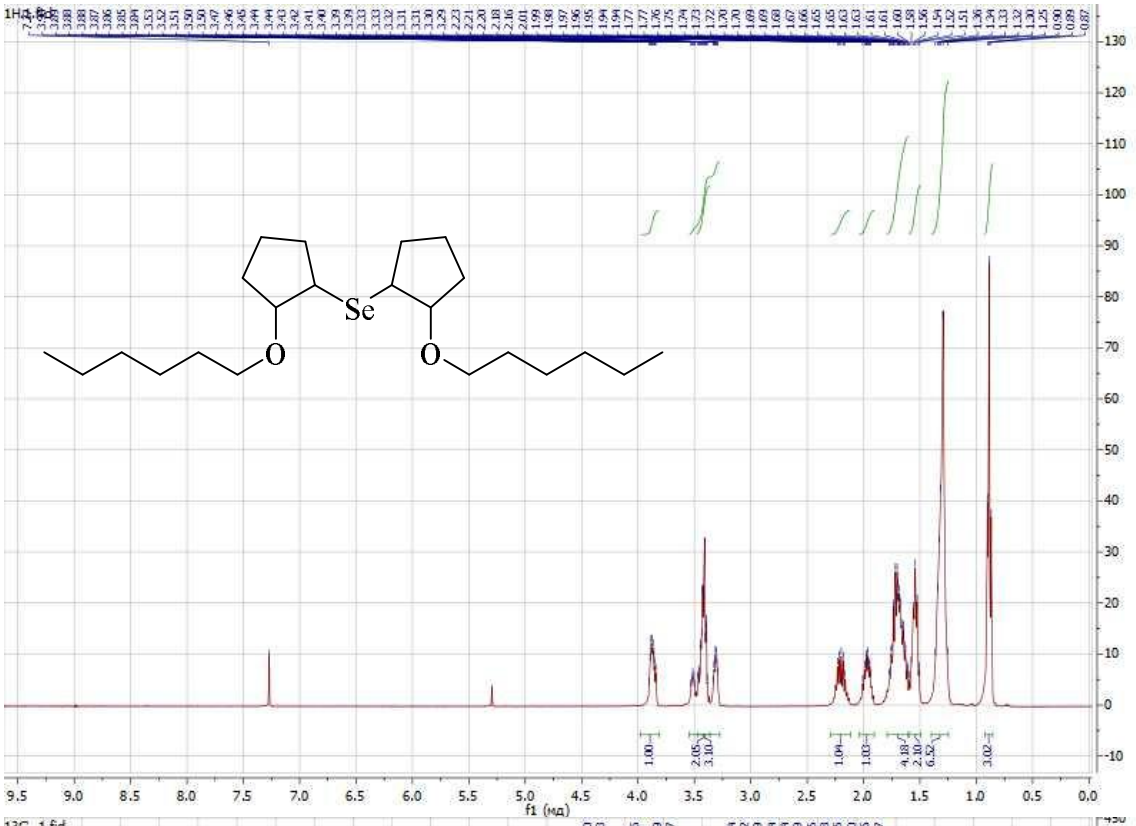


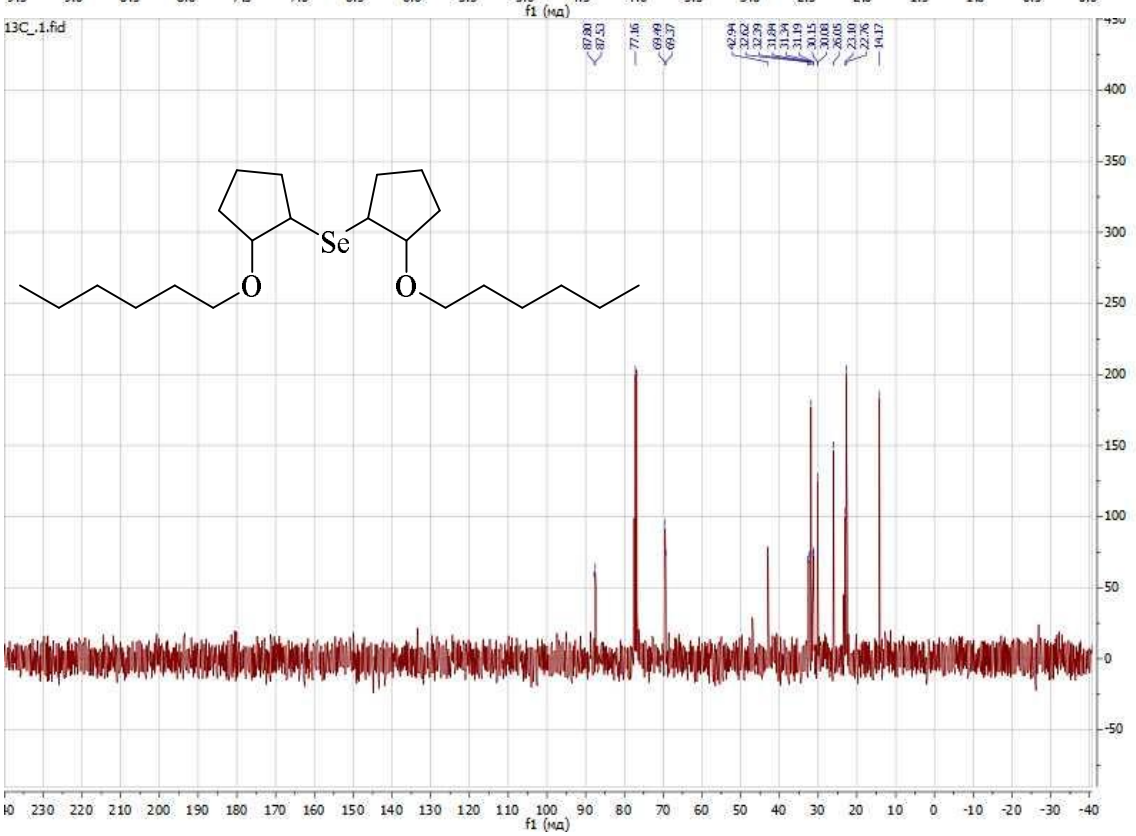


^{1}H- and ^{13}C-NMR spectra of compound 8

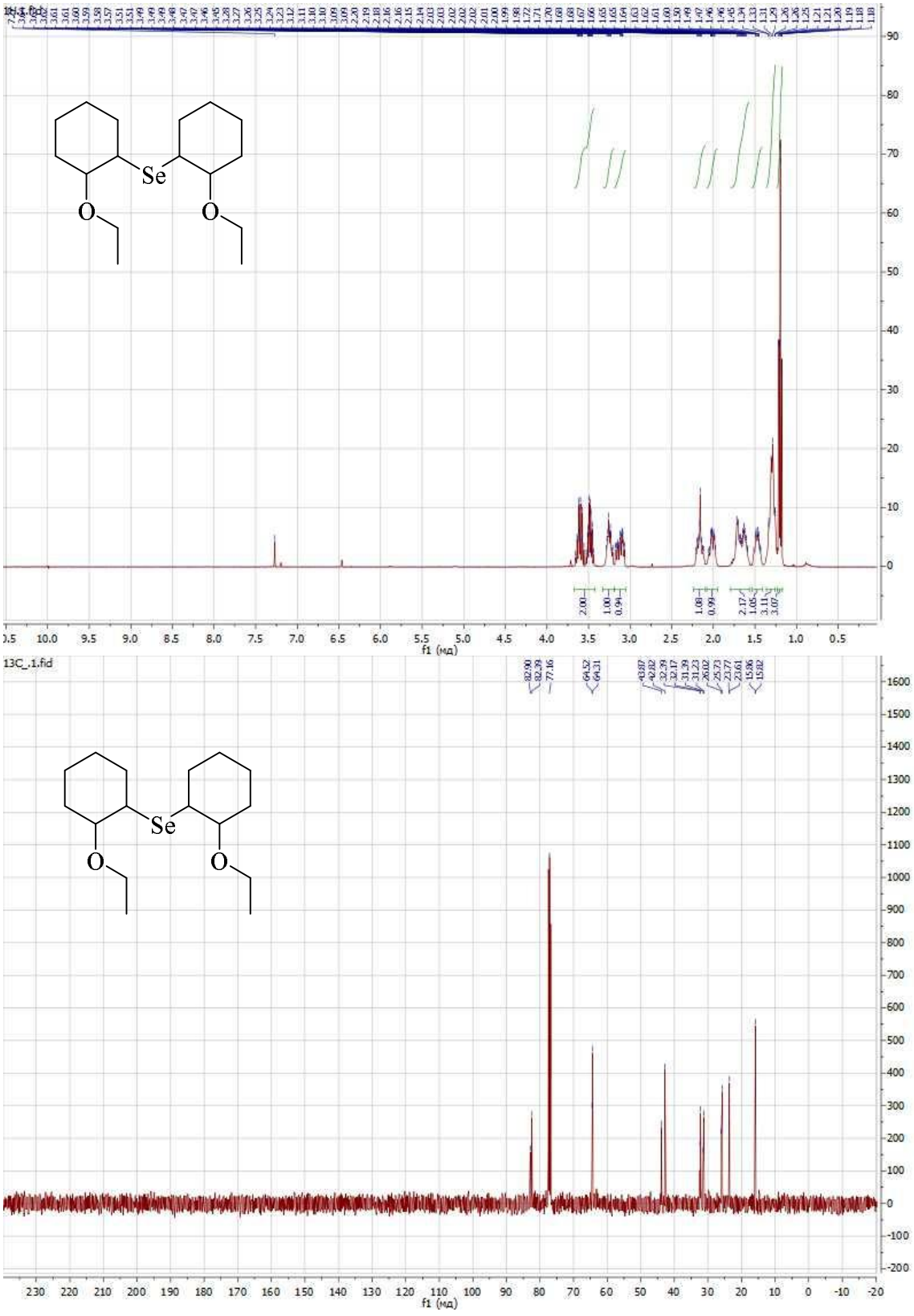


^{1}H- and ^{13}C-NMR spectra of compound 9

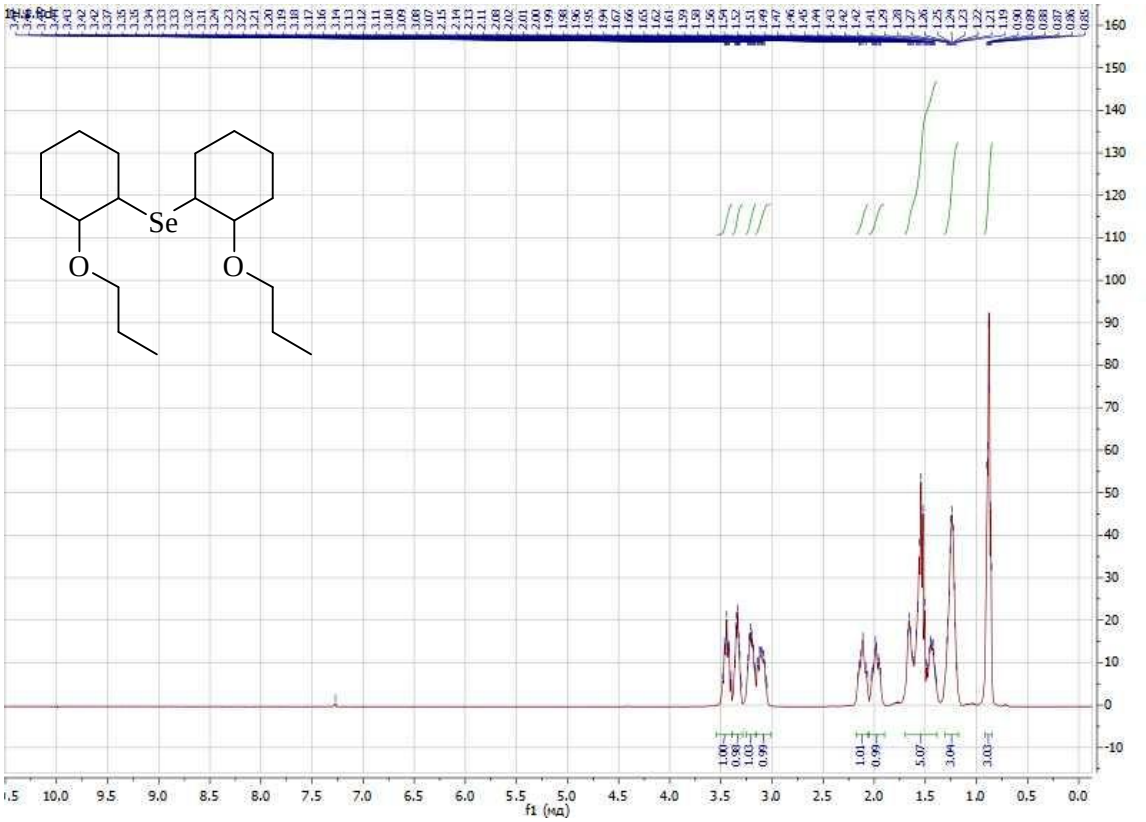


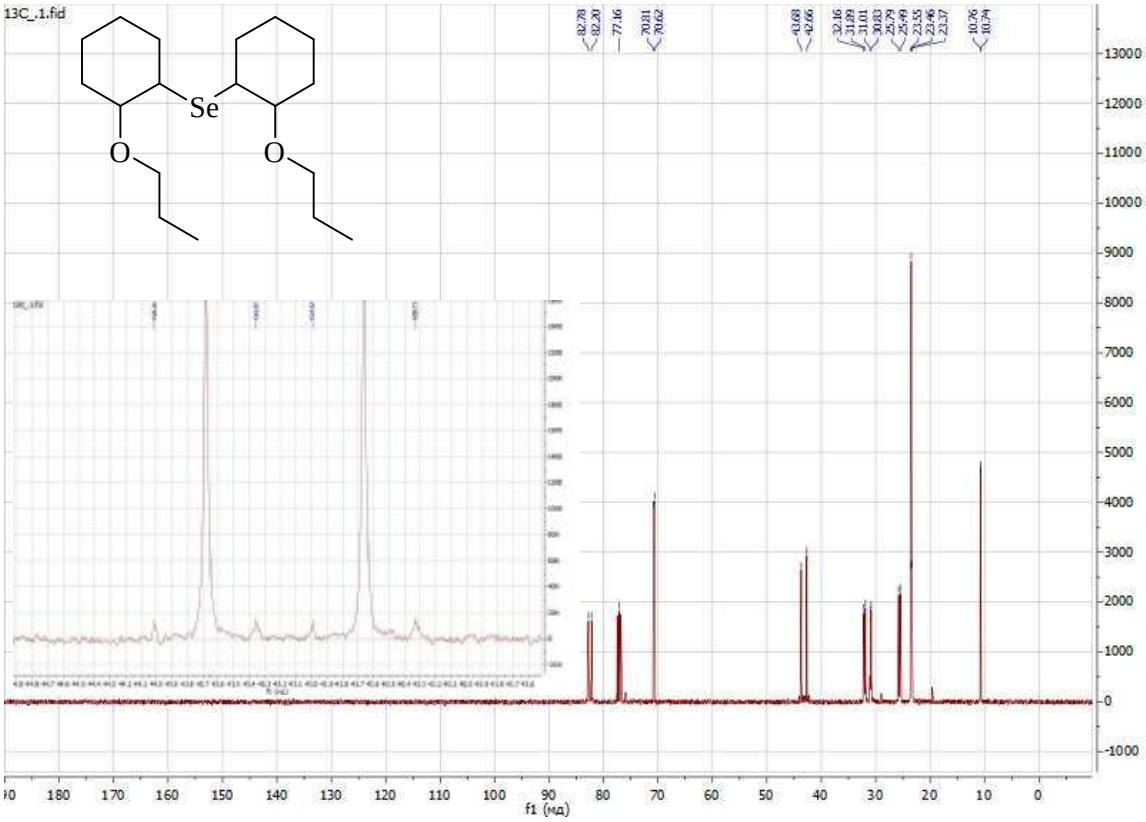


^{1}H- and ^{13}C-NMR spectra of compound 11

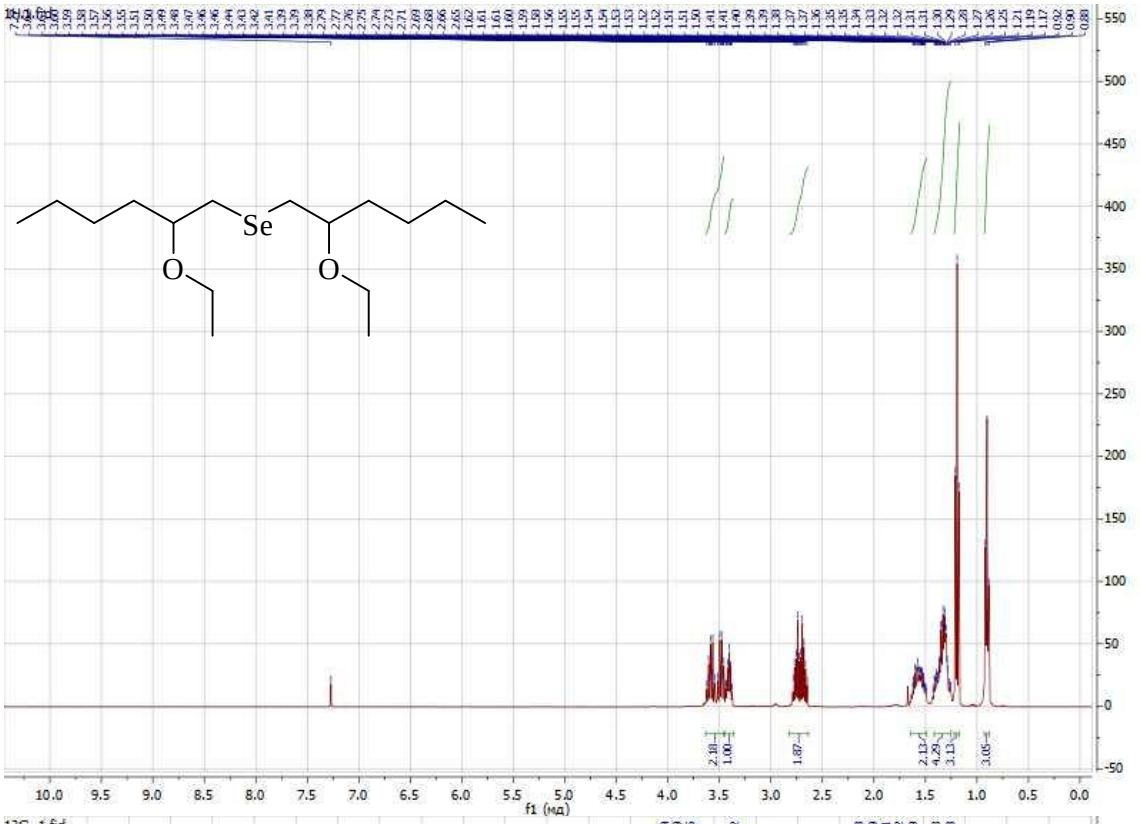


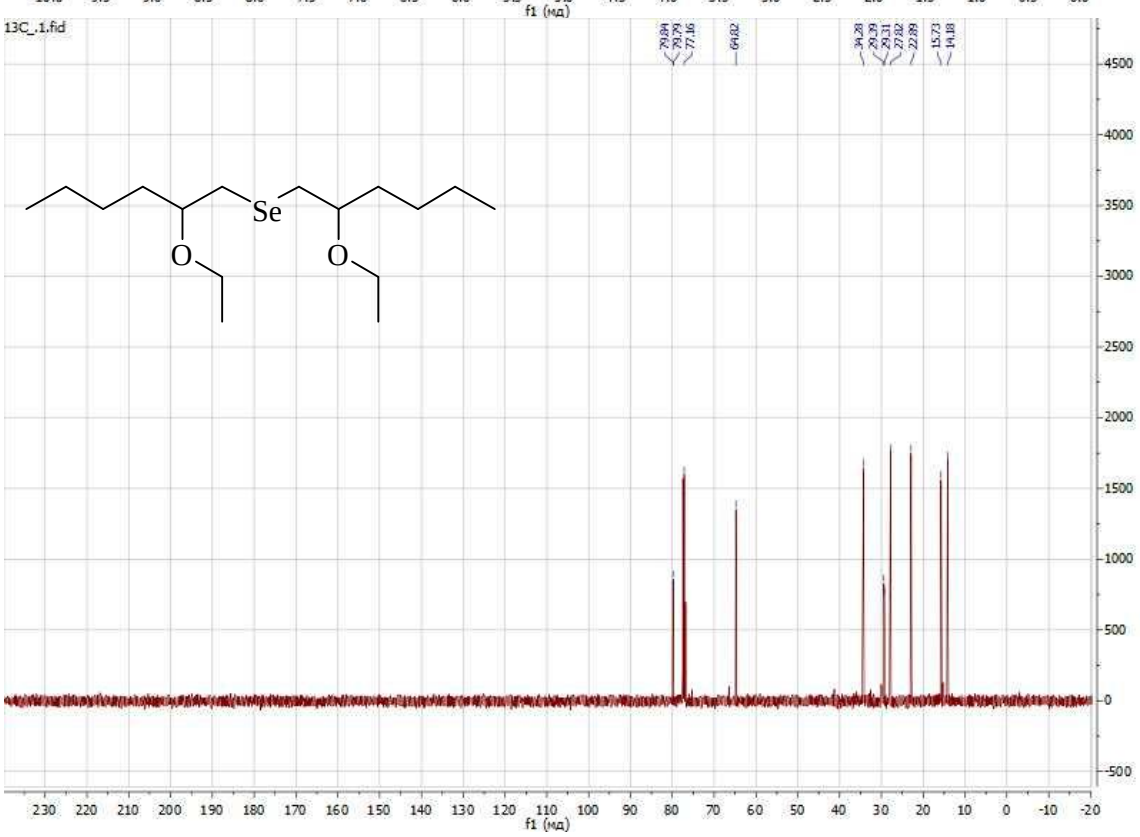
^{1}H- and ^{13}C-NMR spectra of compound 12



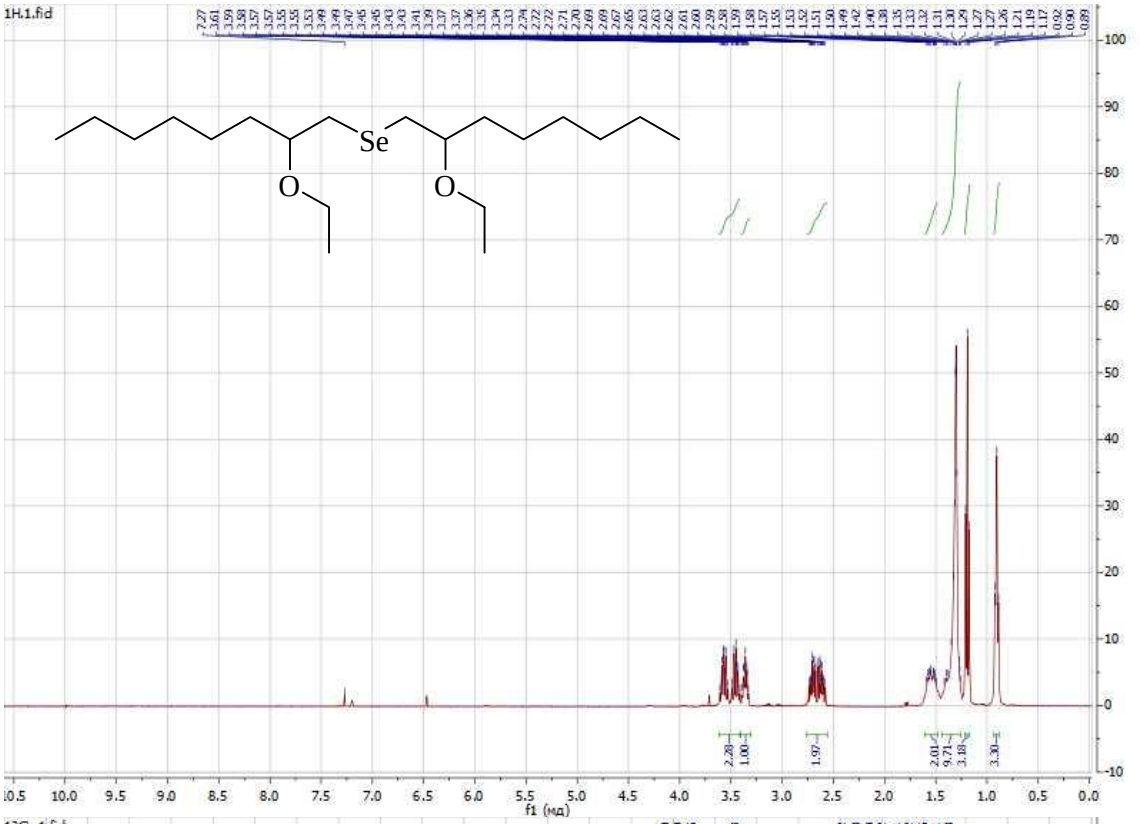


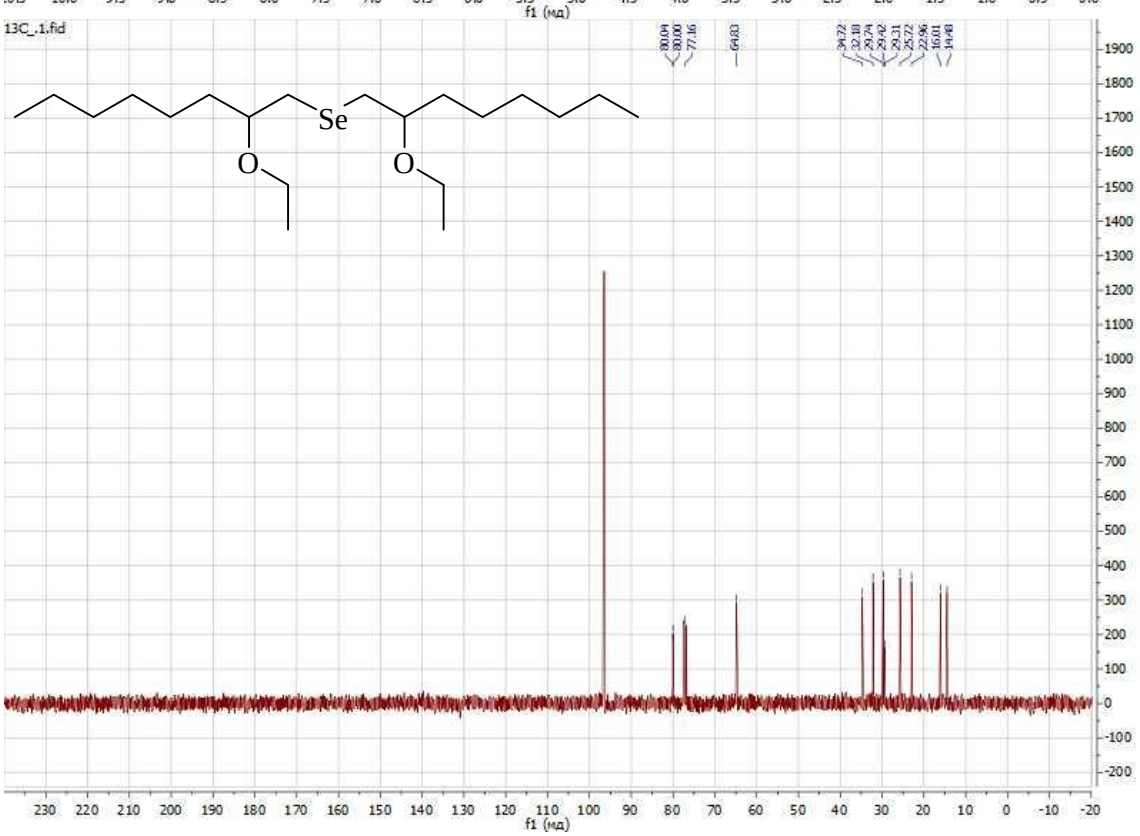
^{1}H- and ^{13}C-NMR spectra of compound 20



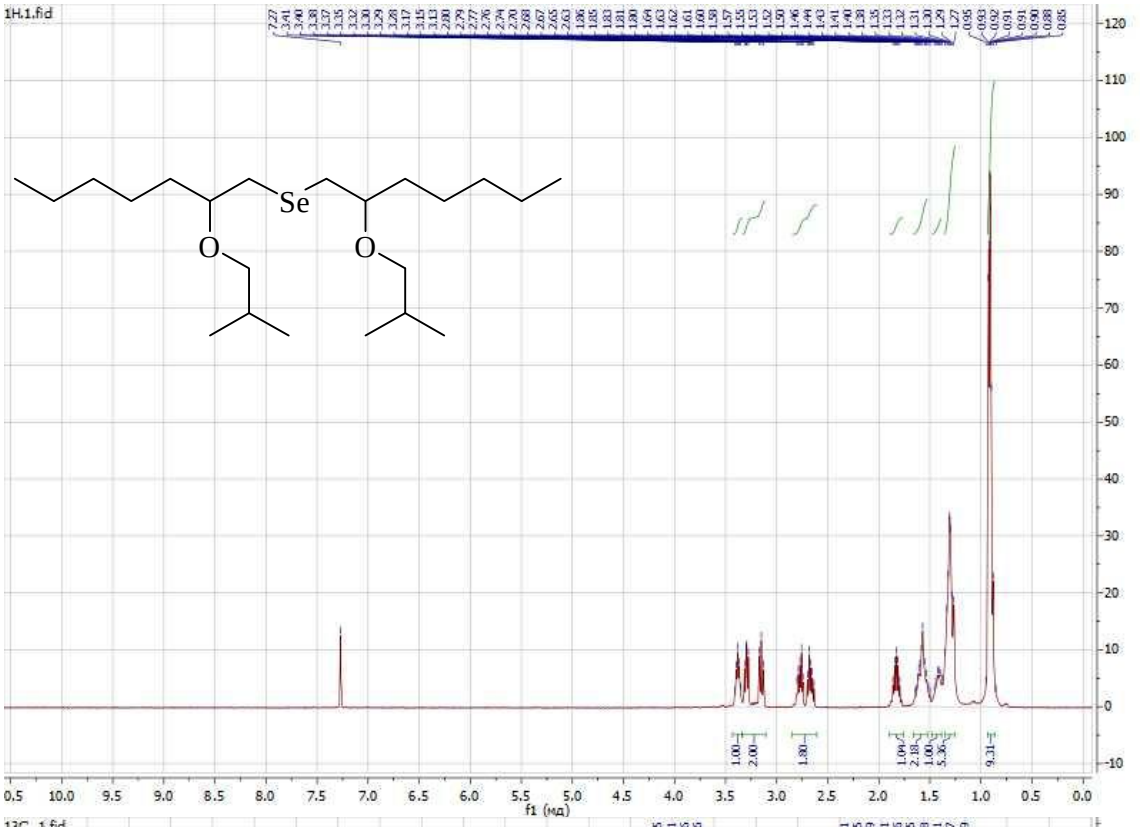


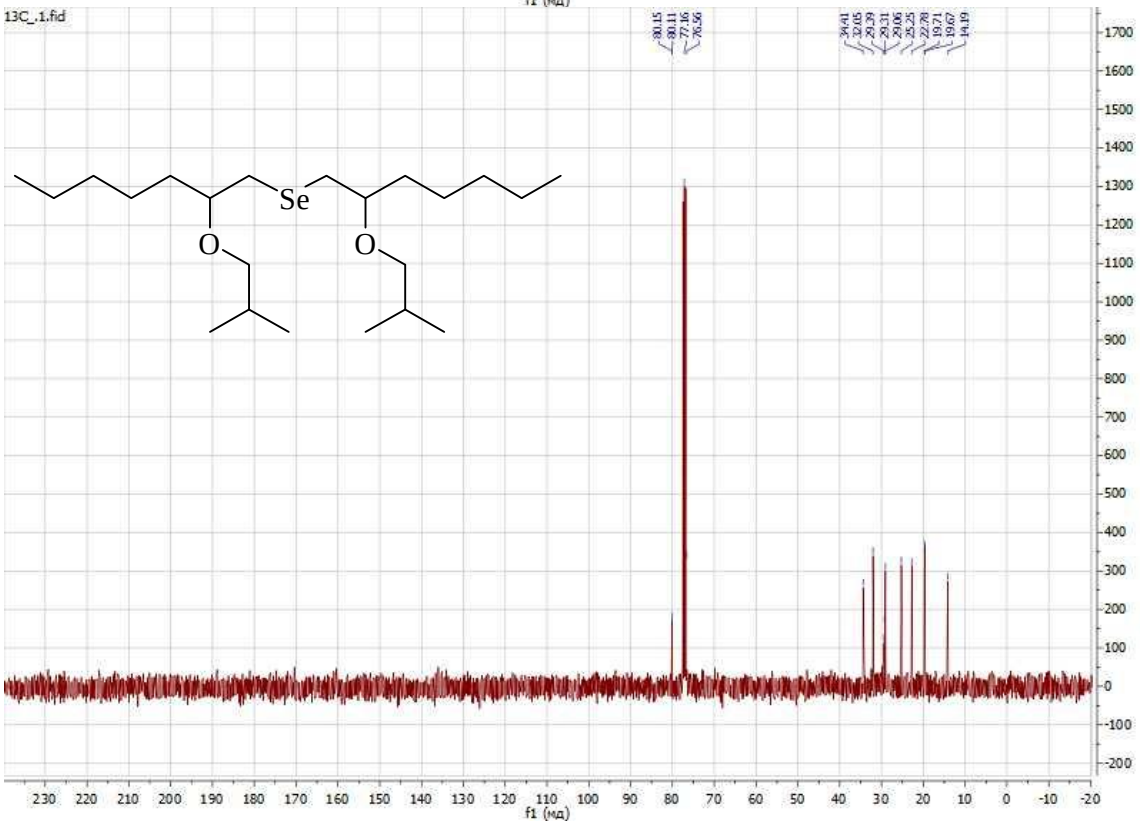
^{1}H- and ^{13}C-NMR spectra of compound 22





^{1}H- and ^{13}C-NMR spectra of compound 23





X-ray crystallographic study of compound 2

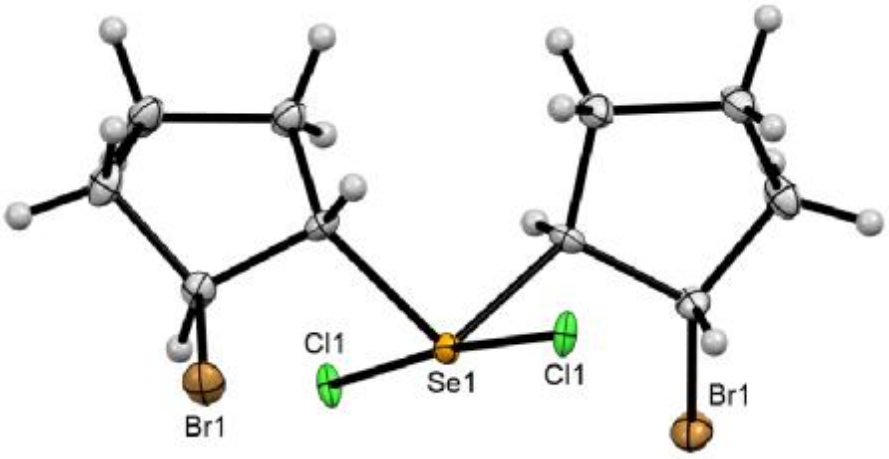


Figure S1. ORTEP plot of compound **2** at 50% thermal ellipsoid probability.

Data were collected on a BRUKER D8 VENTURE PHOTON 100 CMOS diffractometer with MoK_α radiation (λ = 0.71073 Å) using the φ and ω scans technique. Using Olex2 [1], the structure was solved with the ShelXS [2] structure solution program using Direct Methods and refined with the XL [2] refinement package using Least Squares minimisation. Data were corrected for absorption effects using the multi-scan method (SADABS) [3]. All non-hydrogen atoms were refined anisotropically using SHELX [2]. The coordinates of the hydrogen atoms were calculated from geometrical positions.

Crystal data and experimental details are given in Table S1. Selective bond lengths, bond angles and torsion angles are given in Table S2 (Electronic Supplementary Information).

Table S1 contains CCDC reference number of the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk

Table S1. X-ray crystallographic data for compound **2**.

CCDC number	2207651
Empirical formula	$C_{10}H_{16}Cl_2SeBr_2$
Formula weight / g·mol^{-1}	445.91
Crystal system	Monoclinic
Space group	*C2/c*
a / Å	20.687(3)
b / Å	6.1158(8)
c / Å	14.942(2)
α, *β*, *γ* / °	90.00, 133.530(3), 90.00
Volume / Å^3	1370.6(3)
Z	4
Density (calculated) / g·cm^{-3}	2.161
Absorptions coefficient / mm^{-1}	8.928
Radiation (*λ* / Å)	MoKα (0.71073)
Temperature / K	100(2)
2*Θ* range / °	2.72 – 30.07
Crystal size / mm	0.18 × 0.10 × 0.05
Crystal habit	yellow, plate
F(000)	856
Index ranges	$-23 \le h \le 28$, $-8 \le k \le 8$, $-21 \le l \le 20$
Reflections collected	18977
Independent reflections	2009 [R(int) = 0.0841, Rsigma = 0.0493]
Number of ref. parameters	69
R_1 / wR_2 [$I > 2\sigma(I)$]	0.0630 / 0.1667
R_1 / wR_2 (all data)	0.0876 / 0.1768
Goodness-of-fit on F^2	1.136
Completeness [%]	99.8
Largest diff. peak and hole / e·Å^{-3}	3.06/ -1.43
Weight scheme	$w=1/[\sigma^2(F_o^2)+(0.0657P)^2+54.8050P]$ where $P=(F_o^2+2F_c^2)/3$

Table S2. Selective bond lengths, bond angles and torsion angles for compound **2**.

Bond *l*, Å			Angle φ, °				Torsion angle θ, °				
Br1	C1	1.941(7)	Cl1	Se1	Cl11	174.36(10)	Br1	C1	C2	C3	168.6(5)
Se1	Cl11	2.4134(16)	C5	Se1	Cl1	92.4(2)	Br1	C1	C5	Se1	75.6(6)
Se1	Cl1	2.4134(16)	C5	Se1	Cl11	91.2(2)	Br1	C1	C5	C4	-158.1(5)
Se1	C51	1.987(7)	C51	Se1	Cl11	92.4(2)	Cl11	Se1	C5	C1	66.3(5)
Se1	C5	1.987(7)	C51	Se1	Cl1	91.2(2)	Cl1	Se1	C5	C1	-109.4(5)
C1	C2	1.511(11)	C5	Se1	C51	101.5(4)	Cl11	Se1	C5	C4	-53.3(5)
C1	C5	1.516(10)	C2	C1	Br1	114.2(5)	Cl1	Se1	C5	C4	131.1(5)
C2	C3	1.525(12)	C2	C1	C5	103.7(6)	C1	C2	C3	C4	-41.1(8)
C3	C4	1.544(12)	C5	C1	Br1	111.9(5)	C2	C1	C5	Se1	-160.8(5)
C4	C5	1.546(10)	C1	C2	C3	100.0(6)	C2	C1	C5	C4	-34.5(8)
			C2	C3	C4	105.2(7)	C2	C3	C4	C5	20.4(8)
			C3	C4	C5	104.1(6)	C3	C4	C5	Se1	130.7(6)
			C1	C5	Se1	110.3(5)	C3	C4	C5	C1	8.4(8)
			C1	C5	C4	105.0(6)	C51	Se1	C5	C1	158.9(6)
			C4	C5	Se1	116.5(5)	C51	Se1	C5	C4	39.4(5)
							C5	C1	C2	C3	46.5(8)

References

[1] Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
[2] G.M. Sheldrick. A short history of SHELX. Acta Crystallographica Section A Foundations and Advances. – 2008. – A64. – P. 112 – 122. doi.org/10.1107/S0108767307043930
[3] Bruker (2001). SADABS. Bruker AXS Inc., Madison, Wisconsin, USA., (n.d.).