Thermal and photoinduced copper-promoted C-Se bond formation: Synthesis of 2-alkyl-1,2-benzisoselenazol-3(2*H*)-ones and evaluation against *Mycobacterium tuberculosis*

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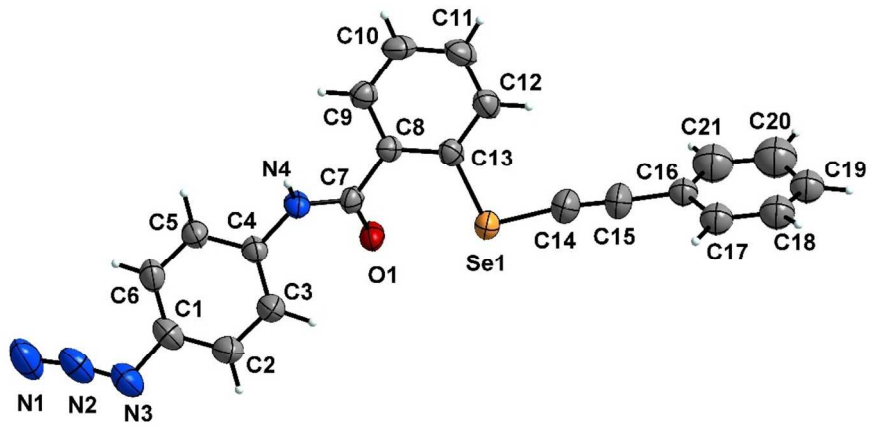
X-Ray crystallographic structure of compound 8



X-ray crystallographic structure of compound **8** (thermal ellipsoid level 50%).

X-ray crystallographic structure of compound **8**

Identification code	Kelly2_TSN1781	
Chemical formula	$C_{21}H_{14}N_4OSe$	
Formula weight	417.32 g/mol	
Temperature	140(2) K	
Wavelength	1.54178 Å	
Crystal size	0.030 x 0.034 x 0.364 mm	
Crystal habit	translucent colorless column	
Crystal system	orthorhombic	
Space group	P c c n	
Unit cell dimensions	a = 17.3904(9) Å	α = 90°
	b = 25.4067(13) Å	β = 90°
	c = 8.3251(5) Å	γ = 90°
Volume	3678.3(3) $Å^3$	

Z	8	
Density (calculated)	1.507 g/cm^3	
Absorption coefficient	2.913 mm^{-1}	
F(000)	1680	
Theta range for data collection	3.08 to 73.22°	
Index ranges	-21<=h<=21, -30<=k<=30, -9<=l<=10	
Reflections collected	40827	
Independent reflections	3595 [R(int) = 0.0541]	
Absorption correction	multi-scan	
Structure solution technique	direct methods	
Structure solution program	SHELXS-97 (Sheldrick, 1990)	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-97 (Sheldrick, 1997)	
Function minimized	$\Sigma\ w(F_o^2 - F_c^2)^2$	
Data / restraints /	3595 / 5 / 298	
Goodness-of-fit on F^2	1.042	
Δ/σ_{max}	0.001	
Final R indices	2829 data; $I>2\sigma(I)$	R1 = 0.0458, wR2 = 0.1116
	all data	R1 = 0.0602, wR2 = 0.1208
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0480P)^2+7.0084P]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.565 and -0.882 $eÅ^{-3}$	
R.M.S. deviation from mean	0.069 $eÅ^{-3}$	

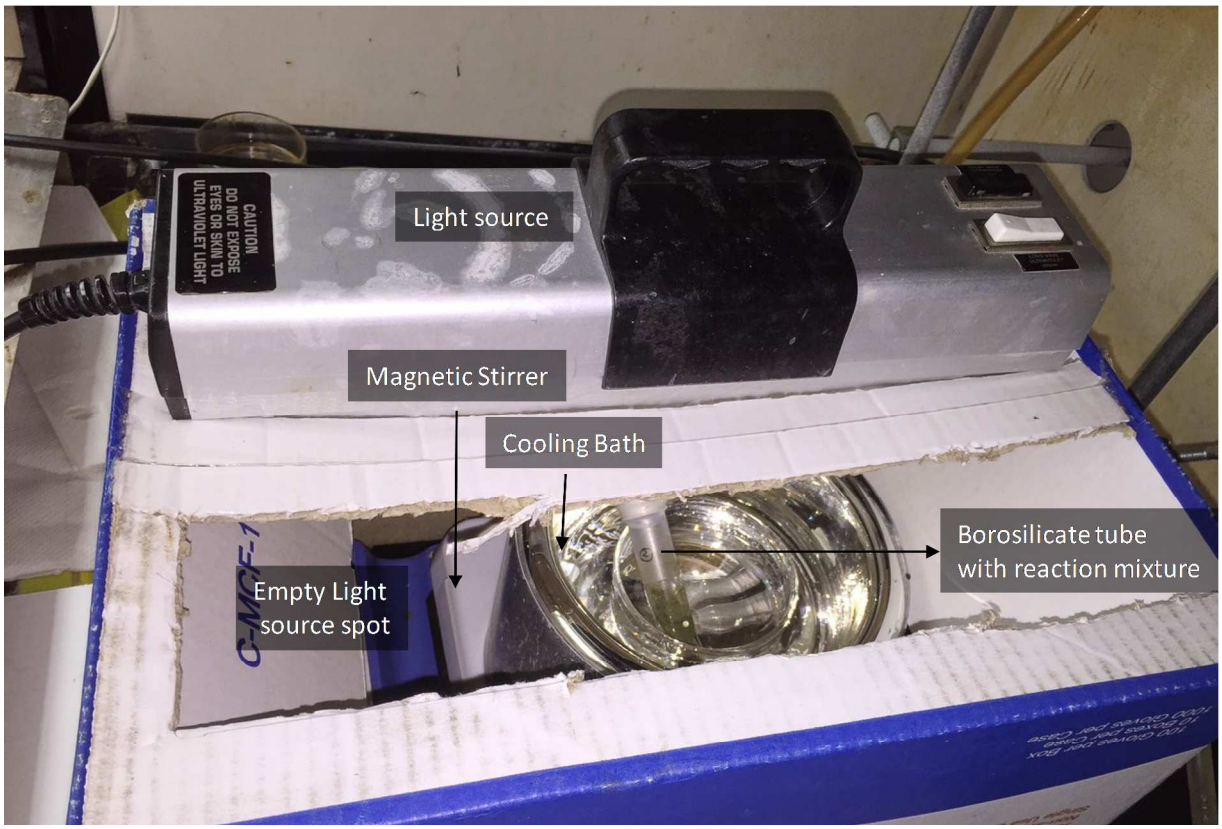


Figure S1. Photochemical reaction setup. The reaction setup consists of cardboard box fitted with two light sources with combined output of 22 Watts 1) 14 watts Rayonet RPR-3000A lamp (spectral energy distribution wavelength range: 250-360 nm), 2) 8 watts Spectronics Corp BLE-8T365 (365nm)).

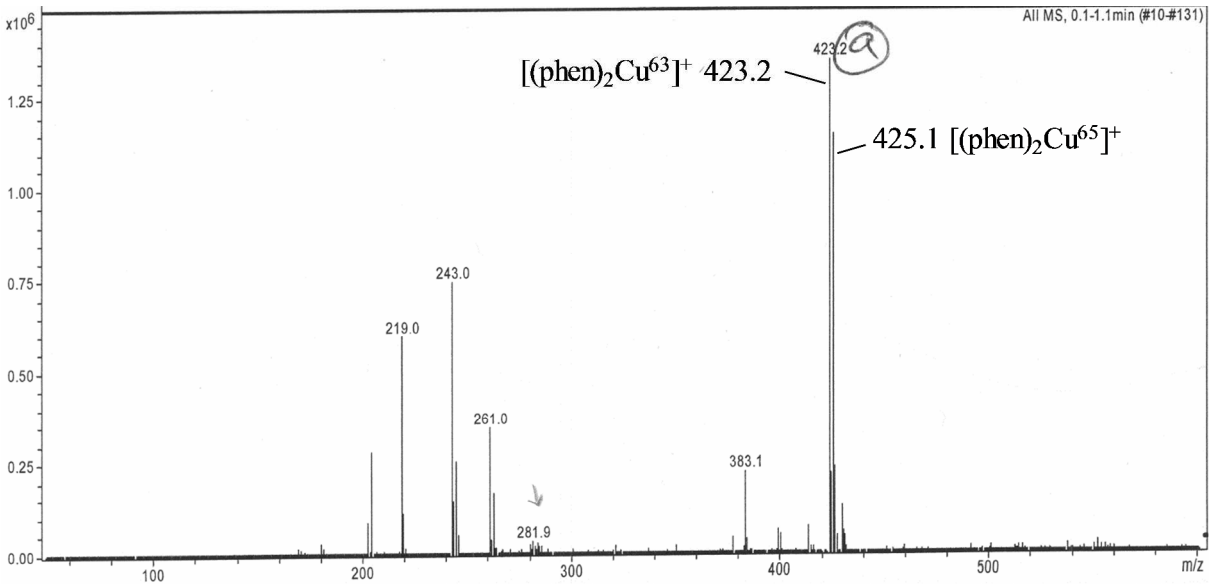


Figure S2. ESI-MS (positive) of $[(phen)_2Cu^{I}]^+$ (**A**) in CH_3CN.

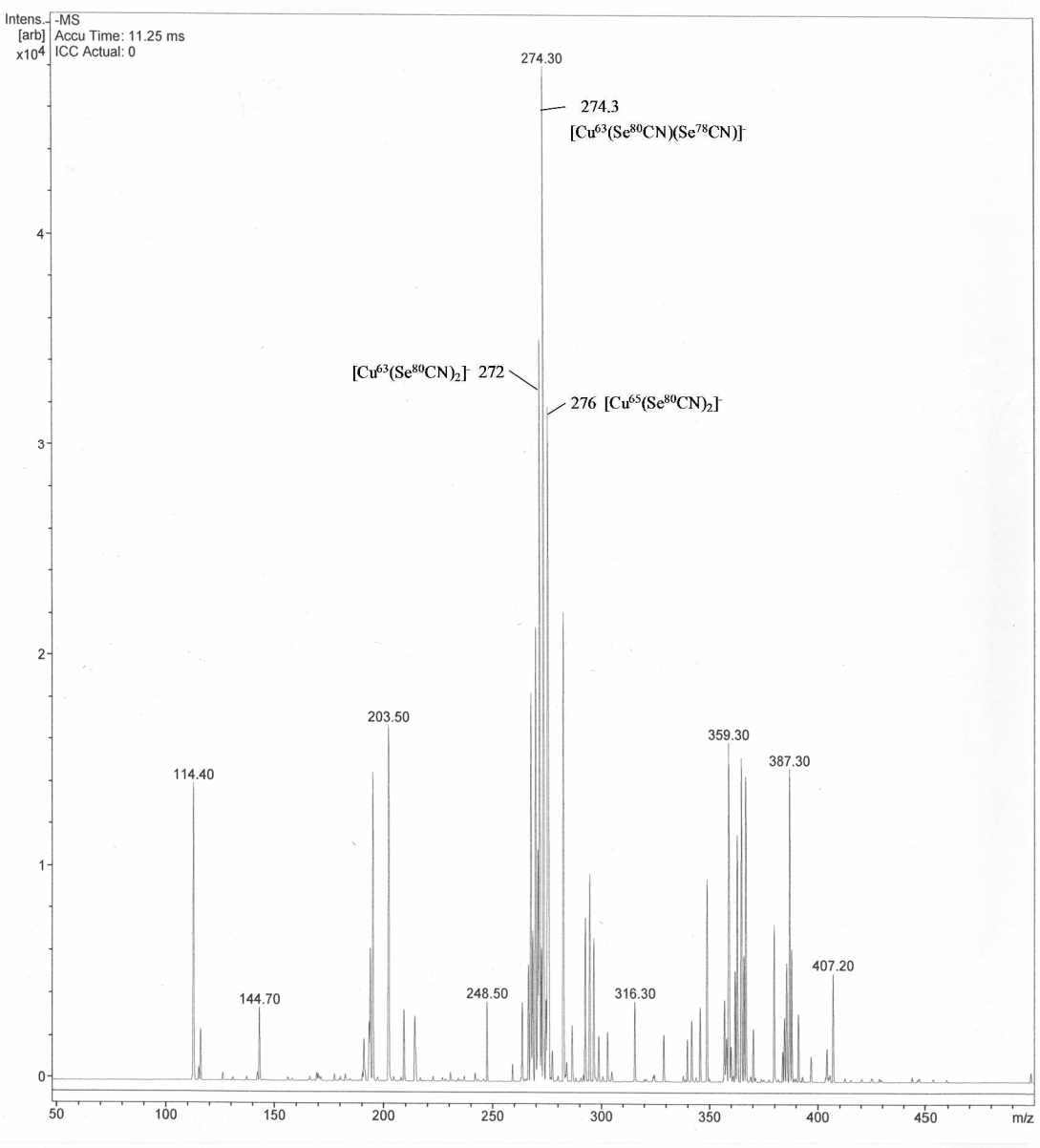


Figure S3. ESI-MS (negative) of $[CuI(SeCN)_2]^-$ (B) in CH_3CN.

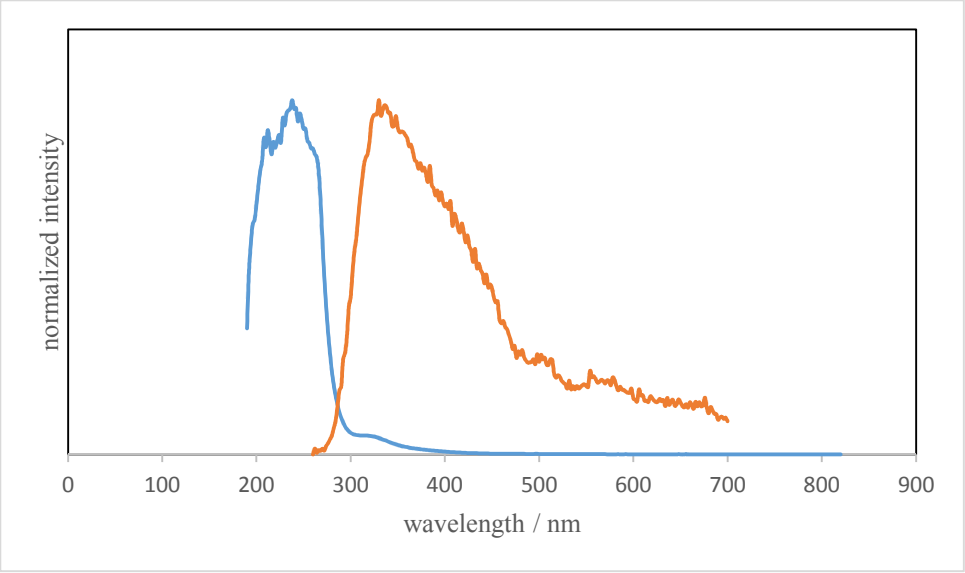


Figure S4. Absorption spectrum of copper complex **B** with λ_{max} = 242 nm (Blue) and emission spectrum at 338 (orange) obtained by excitation copper complex **B** at 242 nm.

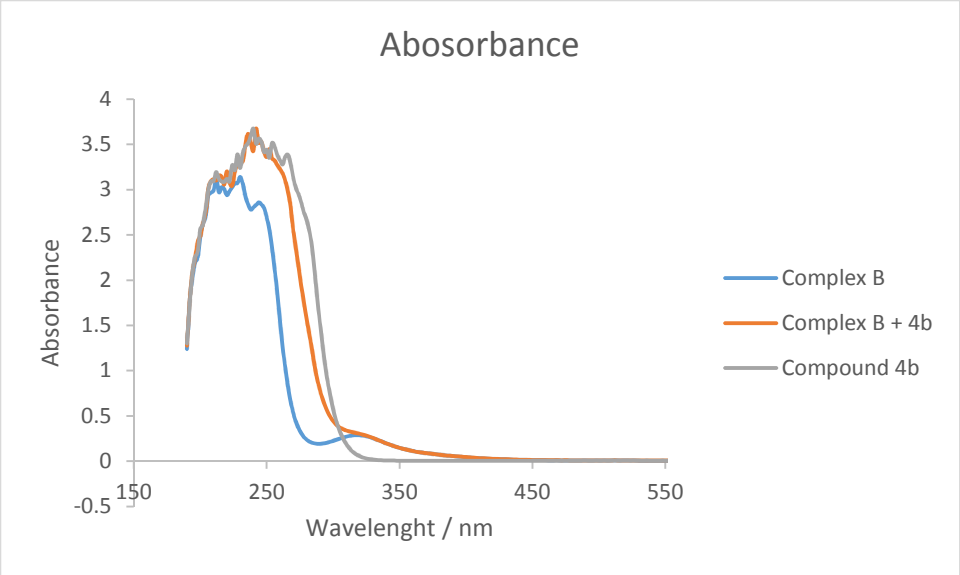


Figure S5. Absorption spectrum of complex **B** (0.91 µM, blue), mixture of complex **B** 0.91 µM)+ **4b** 0.91 µM (orange) and compound **4b** (0.91 µM, grey).

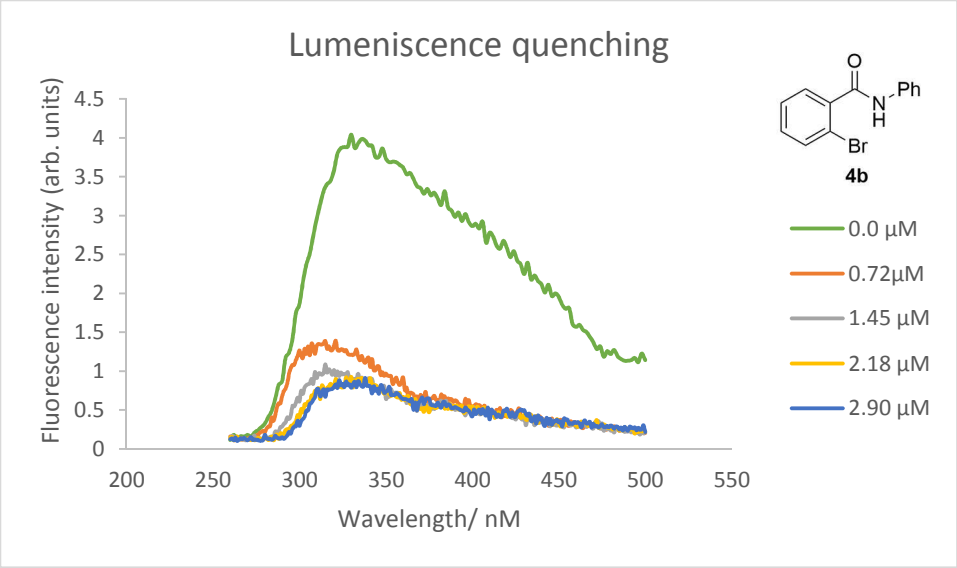
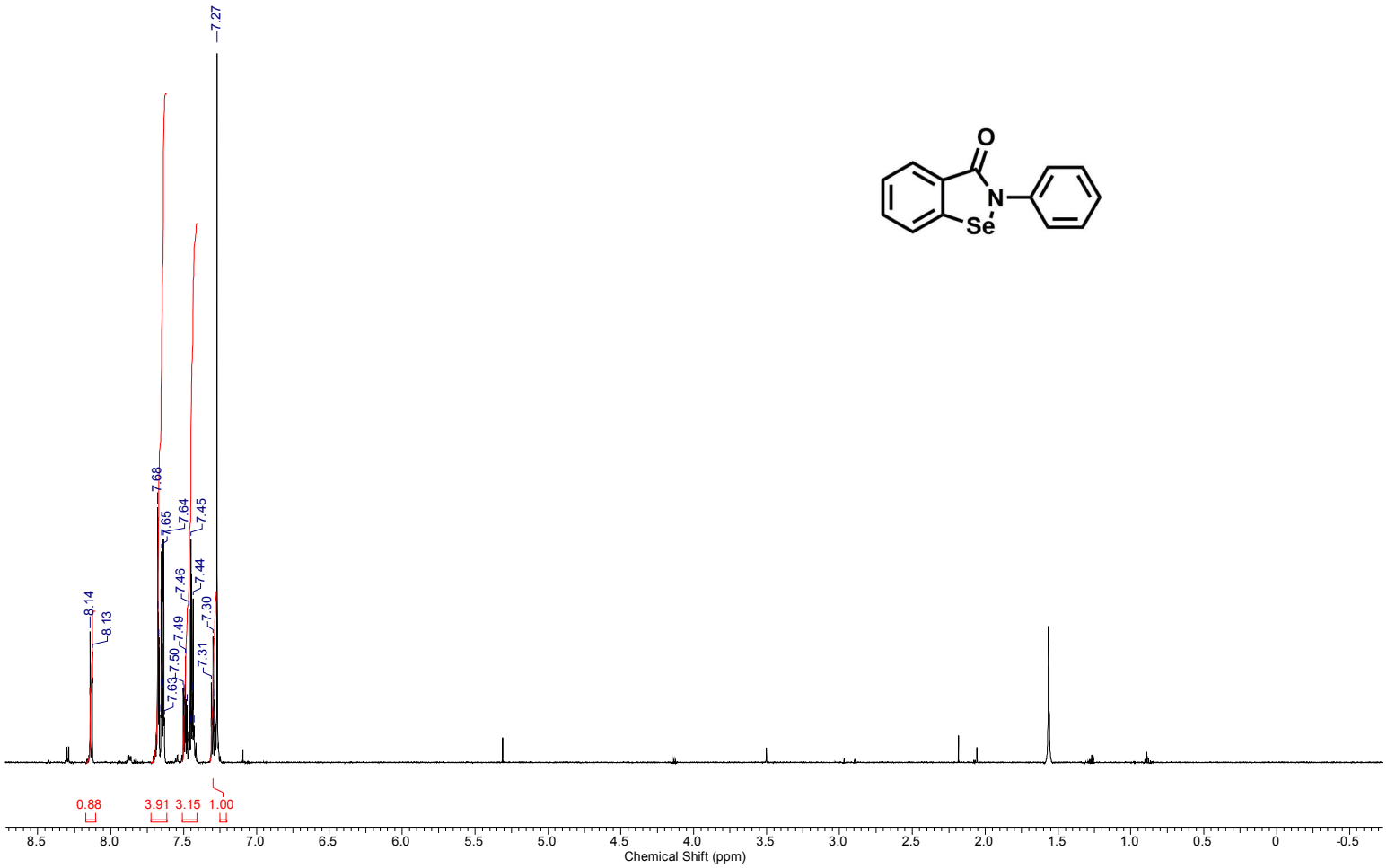


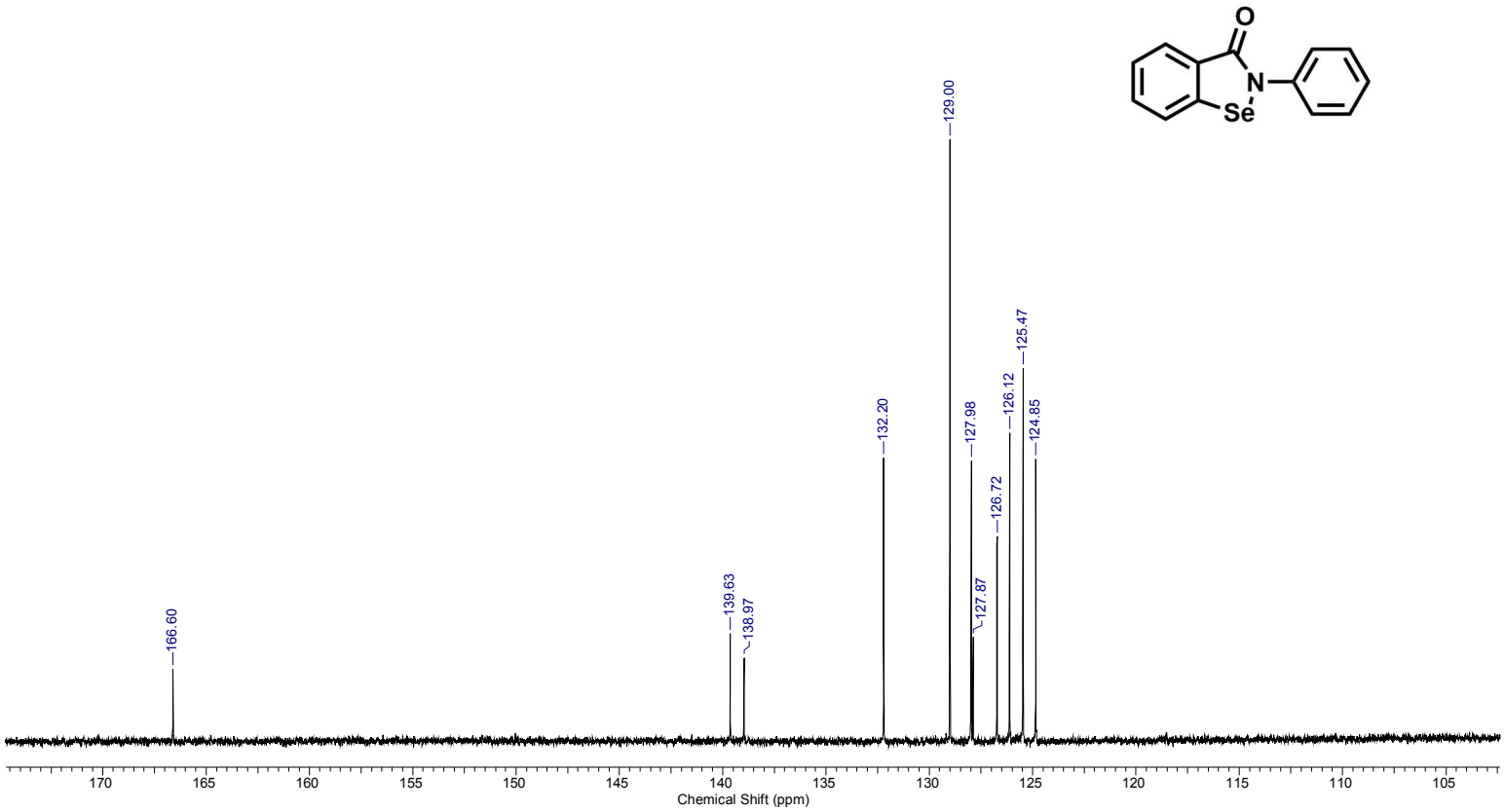
Figure S6. Effect of aryl halide (**4b**) on emission spectra of copper complex **B** (5.25 µM) in acetonitrile. Excitation wavelength: 242

NMR spectra

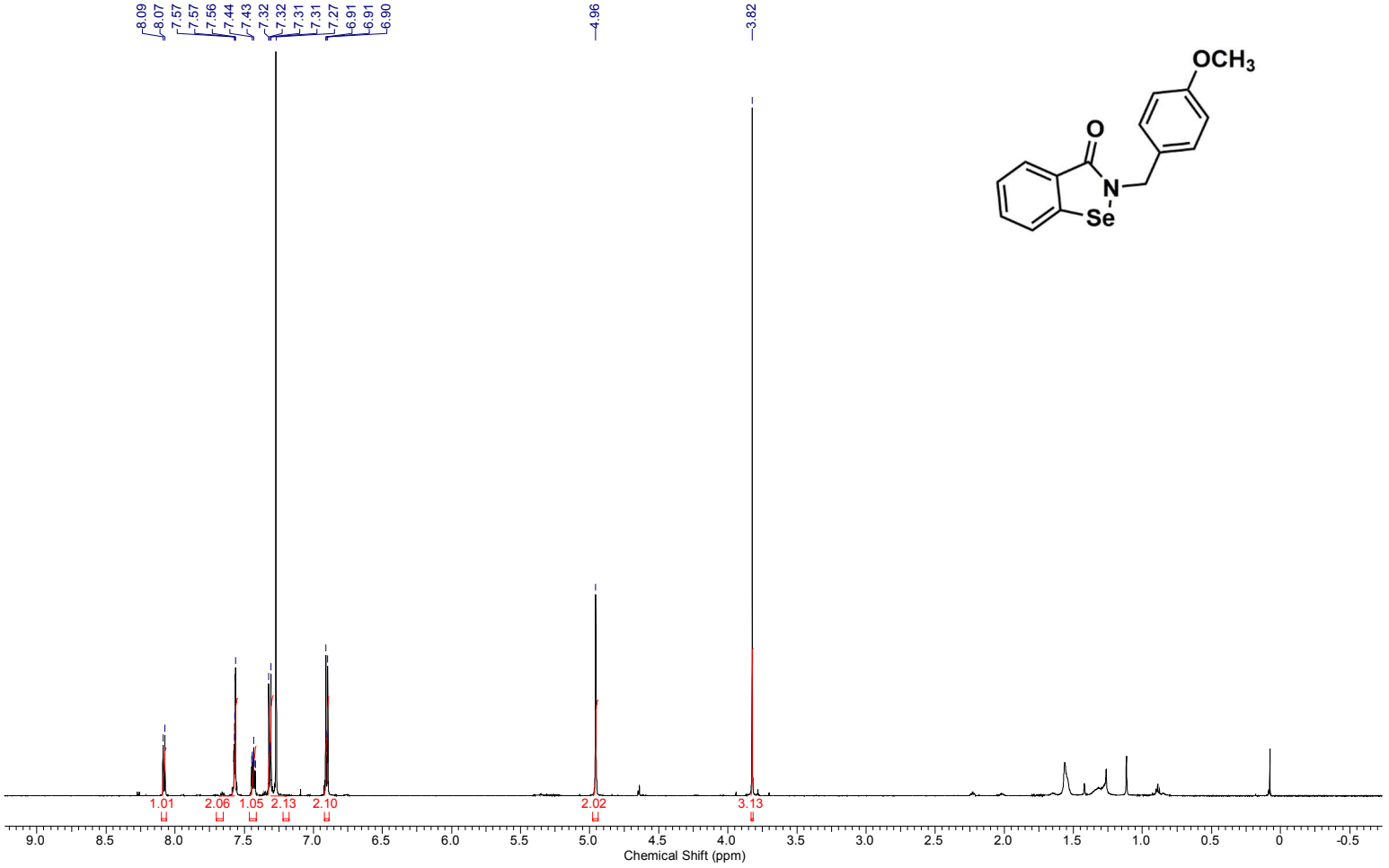
^{1}H-NMR of 2-phenylbenzo[*d*][1,2]selenazol-3(2*H*)-one (1a) (600MHz, $CDCl_3$)



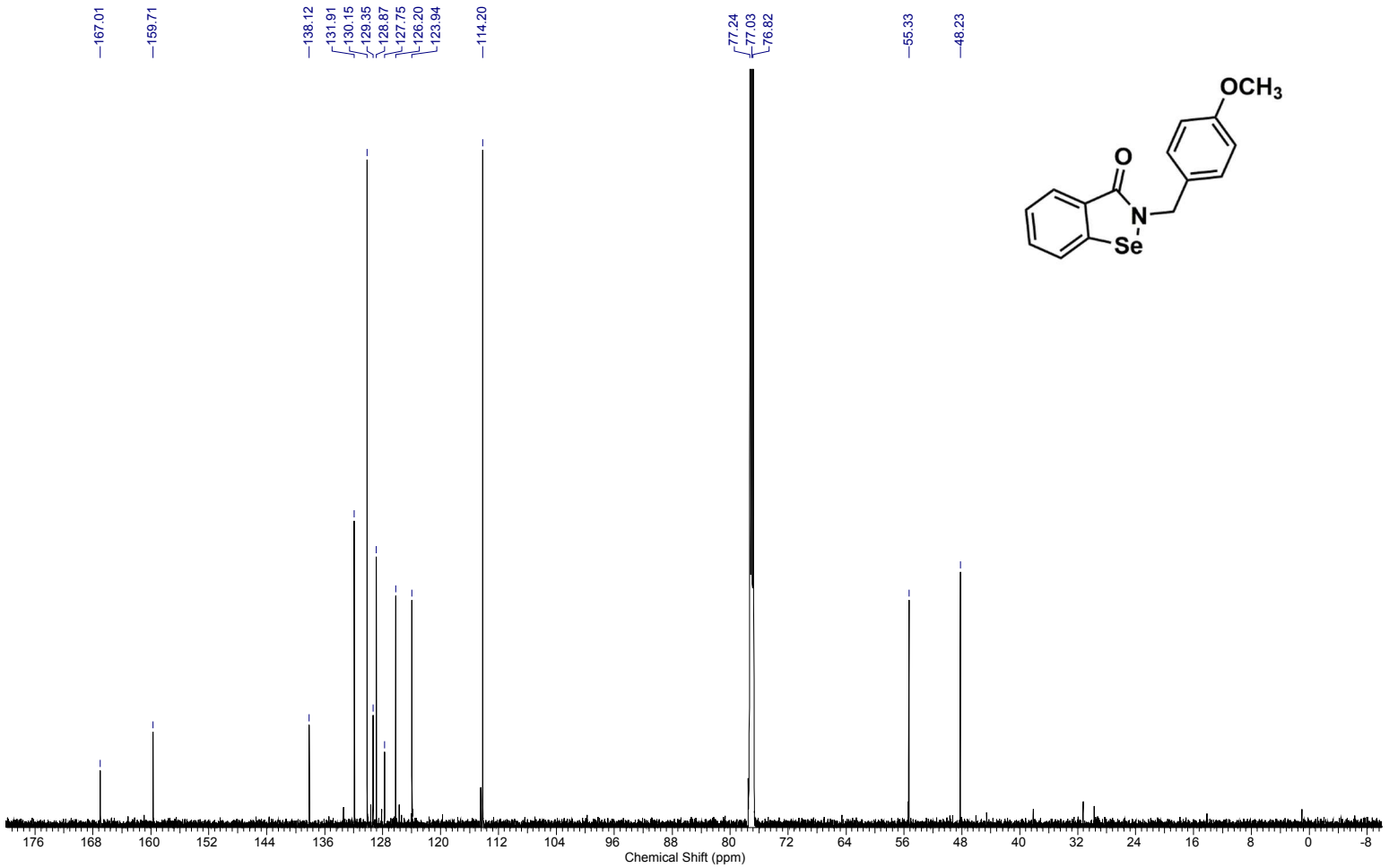
13C-NMR of 2-phenylbenzo[*d*][1,2]selenazol-3(2*H*)-one (1a) (600MHz, MeOD)



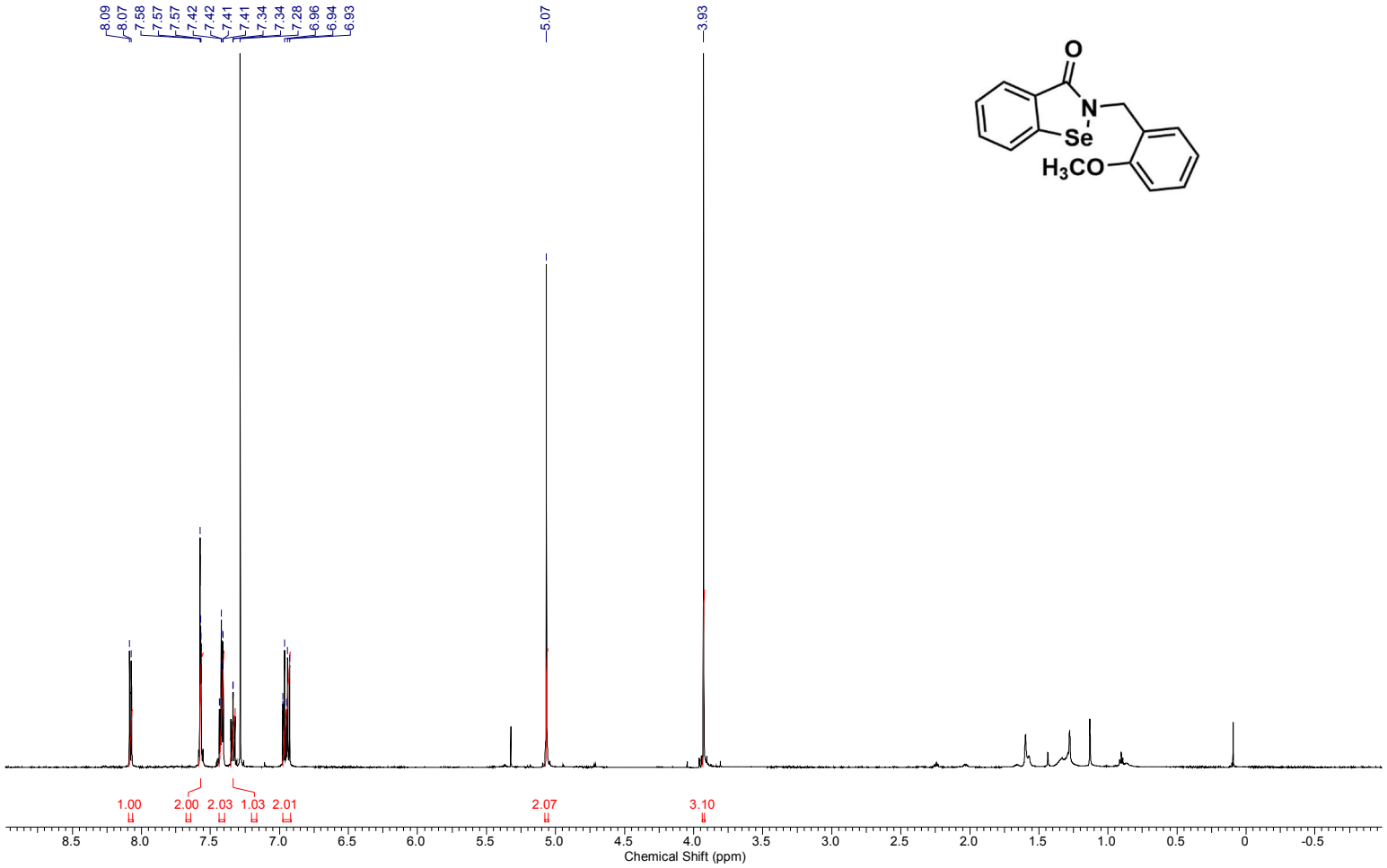
^{1}H-NMR of 2-(4-methoxybenzyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1b)



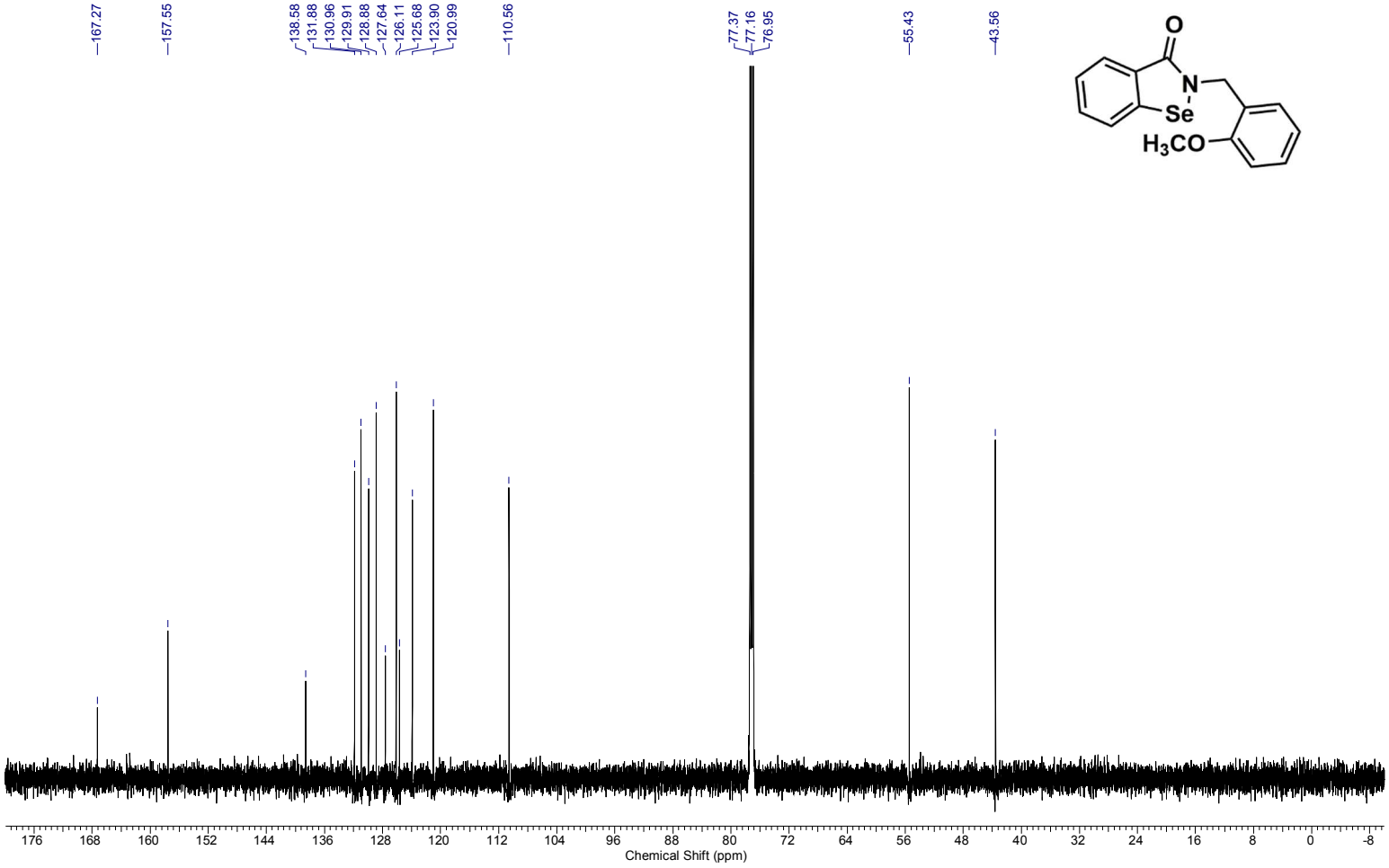
^{13}C-NMR of 2-(4-methoxybenzyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1b)



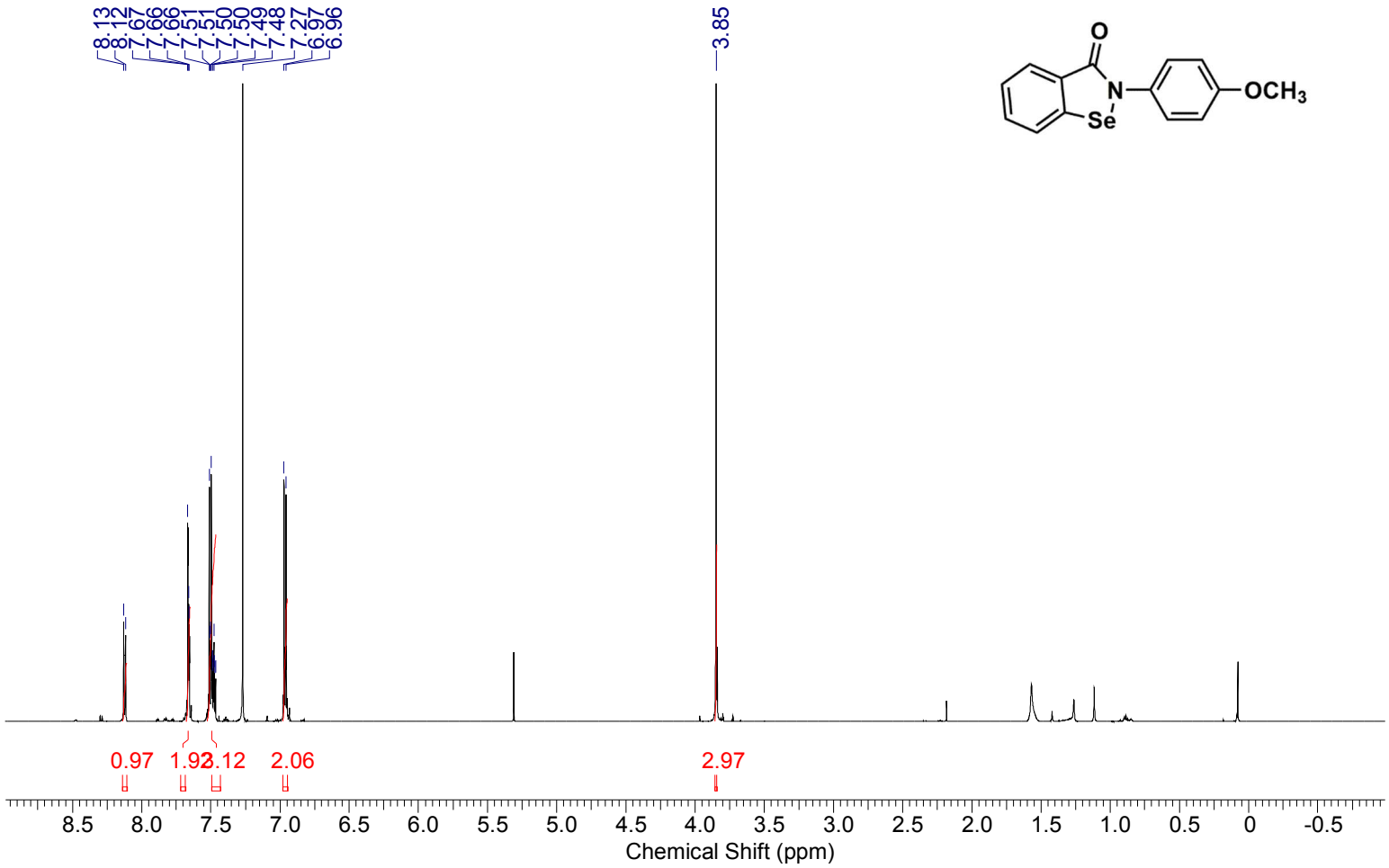
^{1}H-NMR of 2-(2-methoxybenzyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1c)



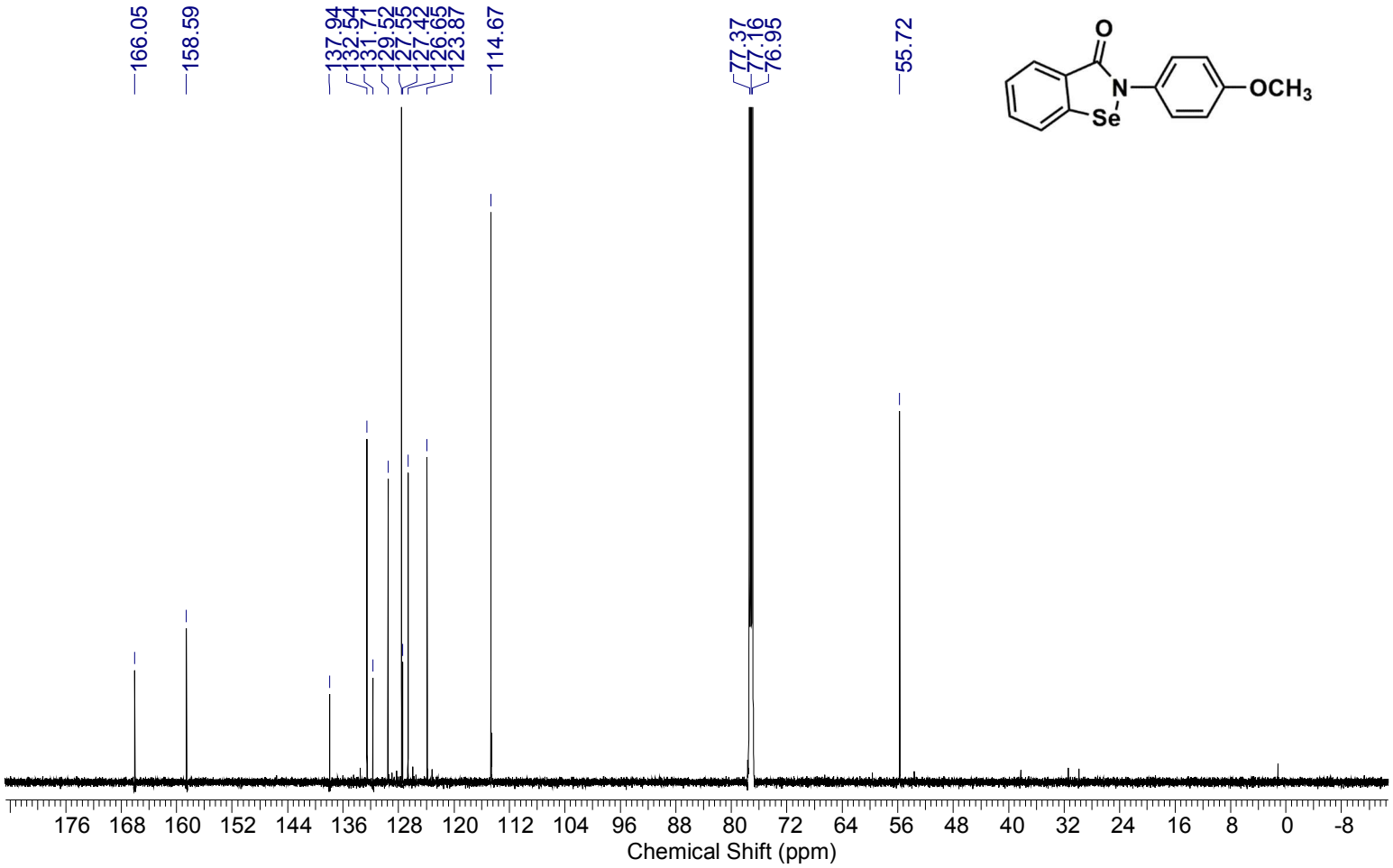
^{13}C-NMR of 2-(2-methoxybenzyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1c)



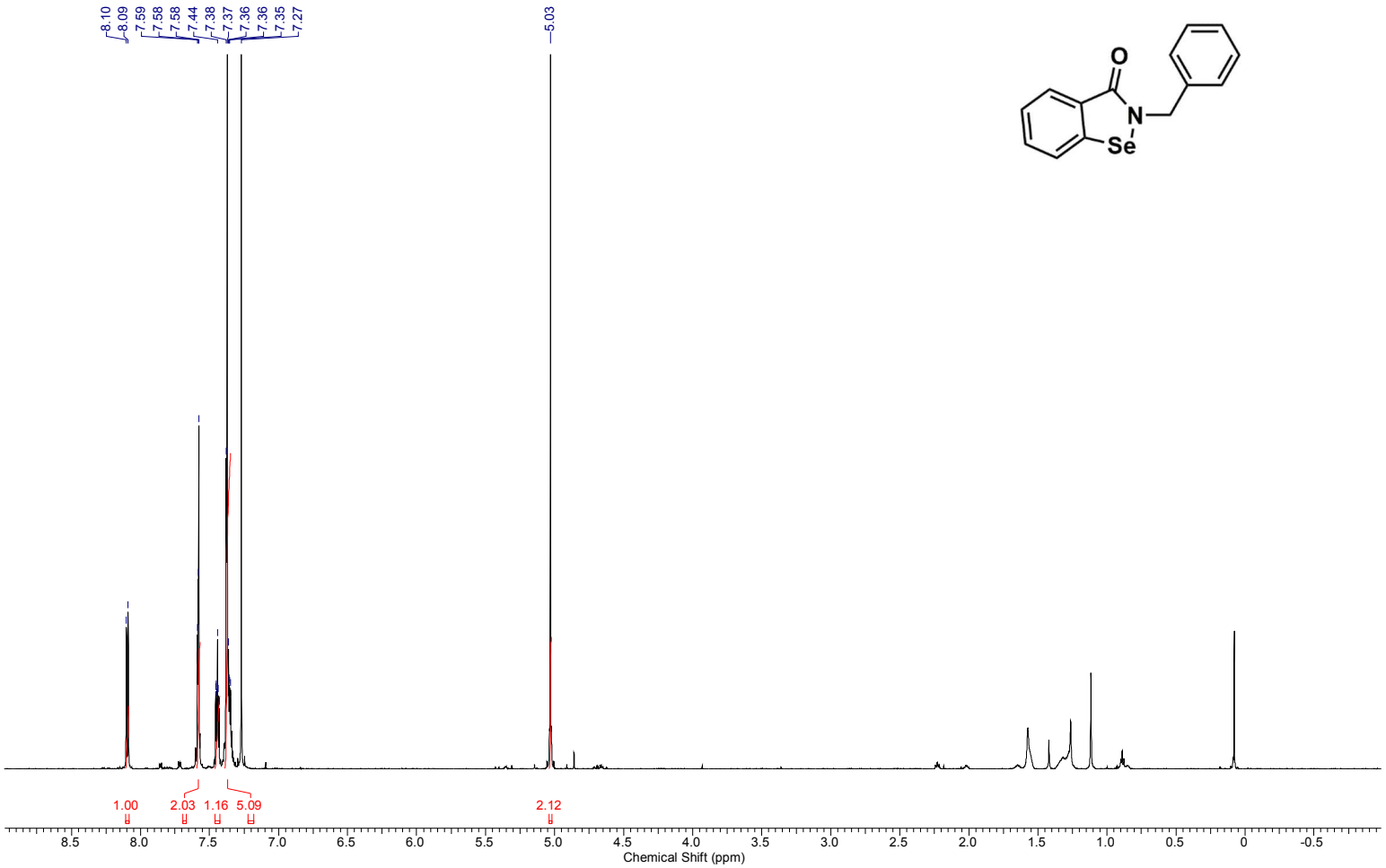
^{1}H-NMR of 2-(4-methoxyphenyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1d)



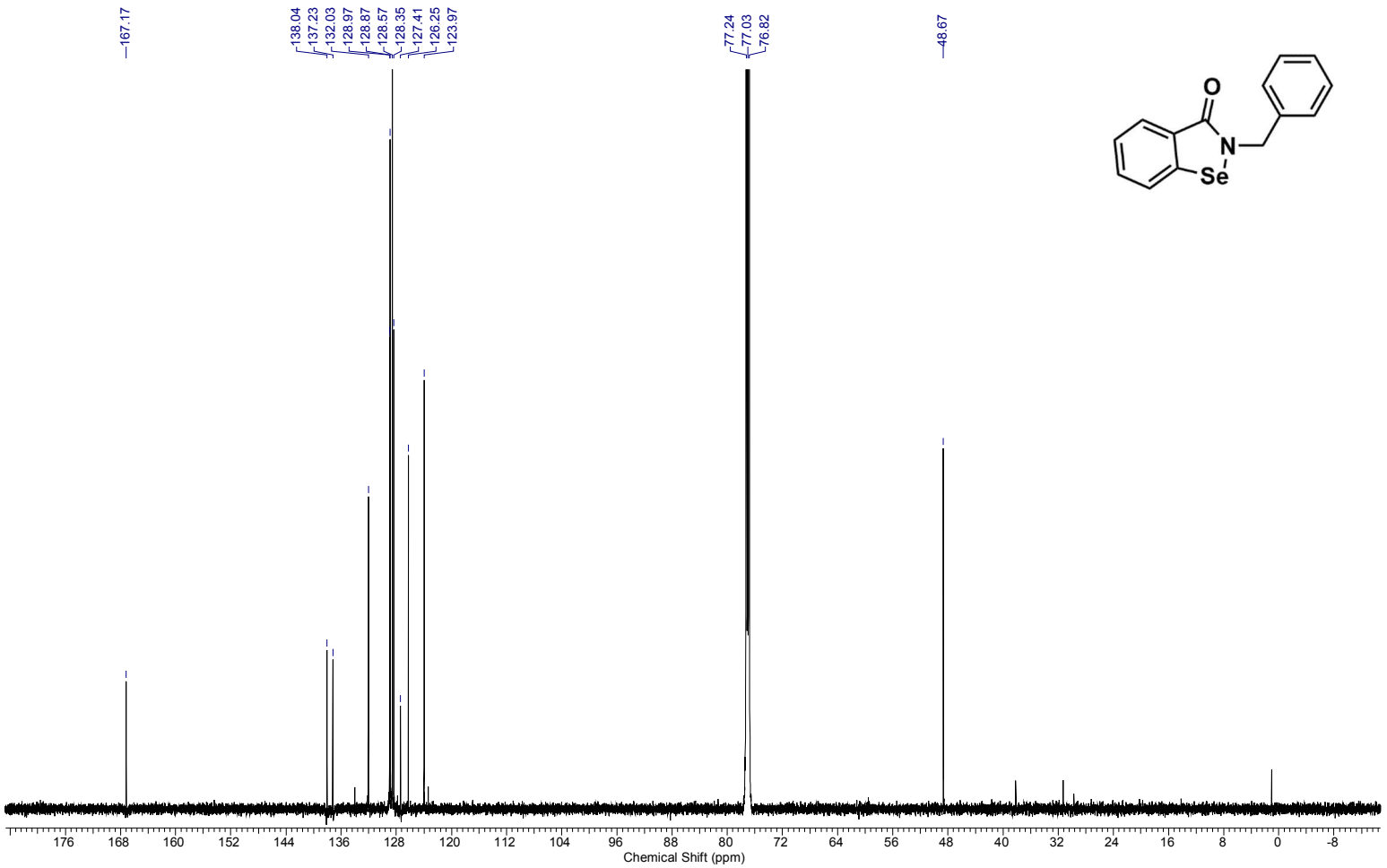
^{13}C-NMR of 2-(4-methoxyphenyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1d)



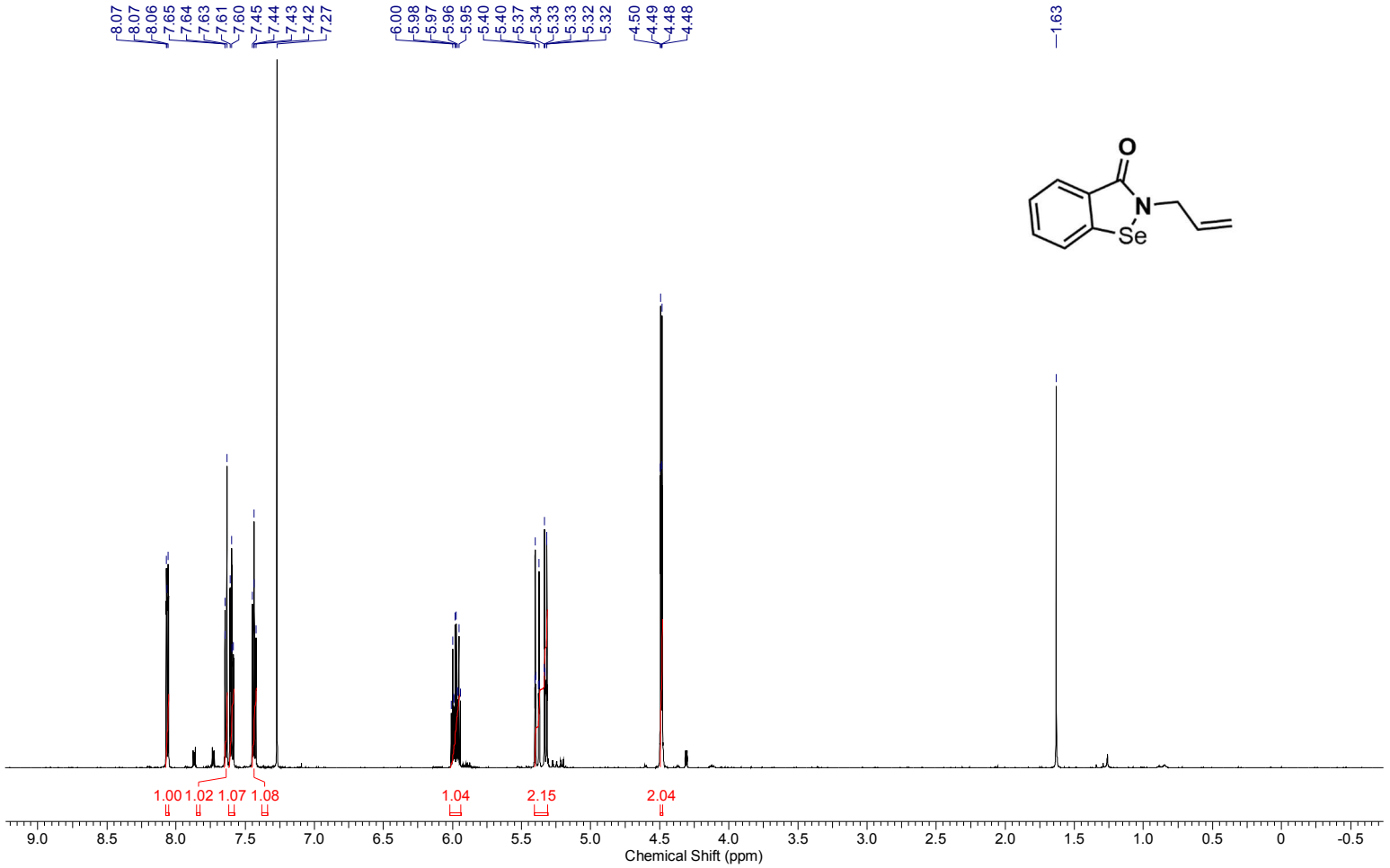
^{1}H-NMR of 2-benzylbenzo[*d*][1,2]selenazol-3(2*H*)-one (1e)



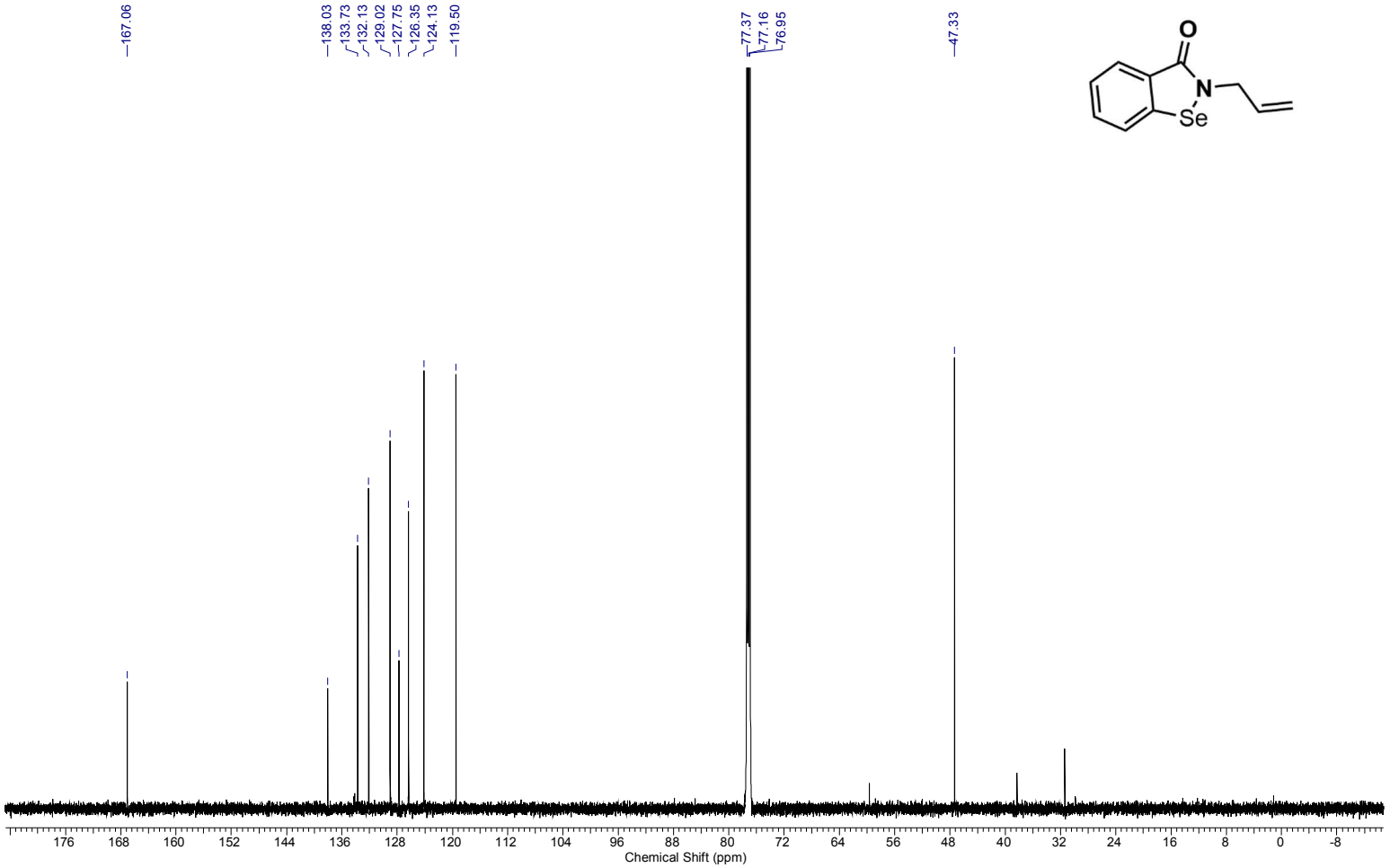
^{13}C-NMR of 2-benzylbenzo[*d*][1,2]selenazol-3(2*H*)-one (1e)



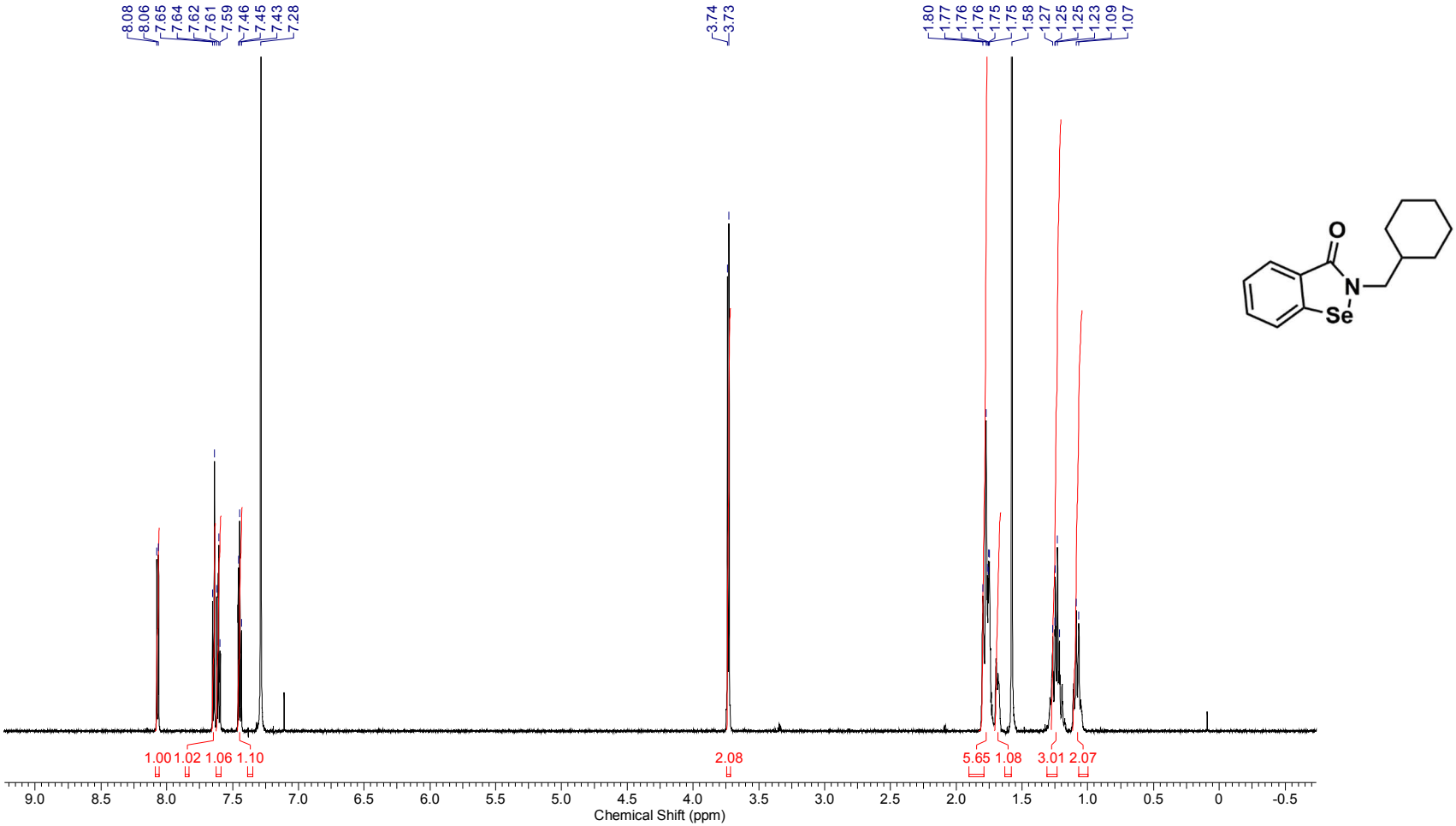
[1]H-NMR of 2-allylbenzo[*d*][1,2]selenazol-3(2*H*)-one (1f)



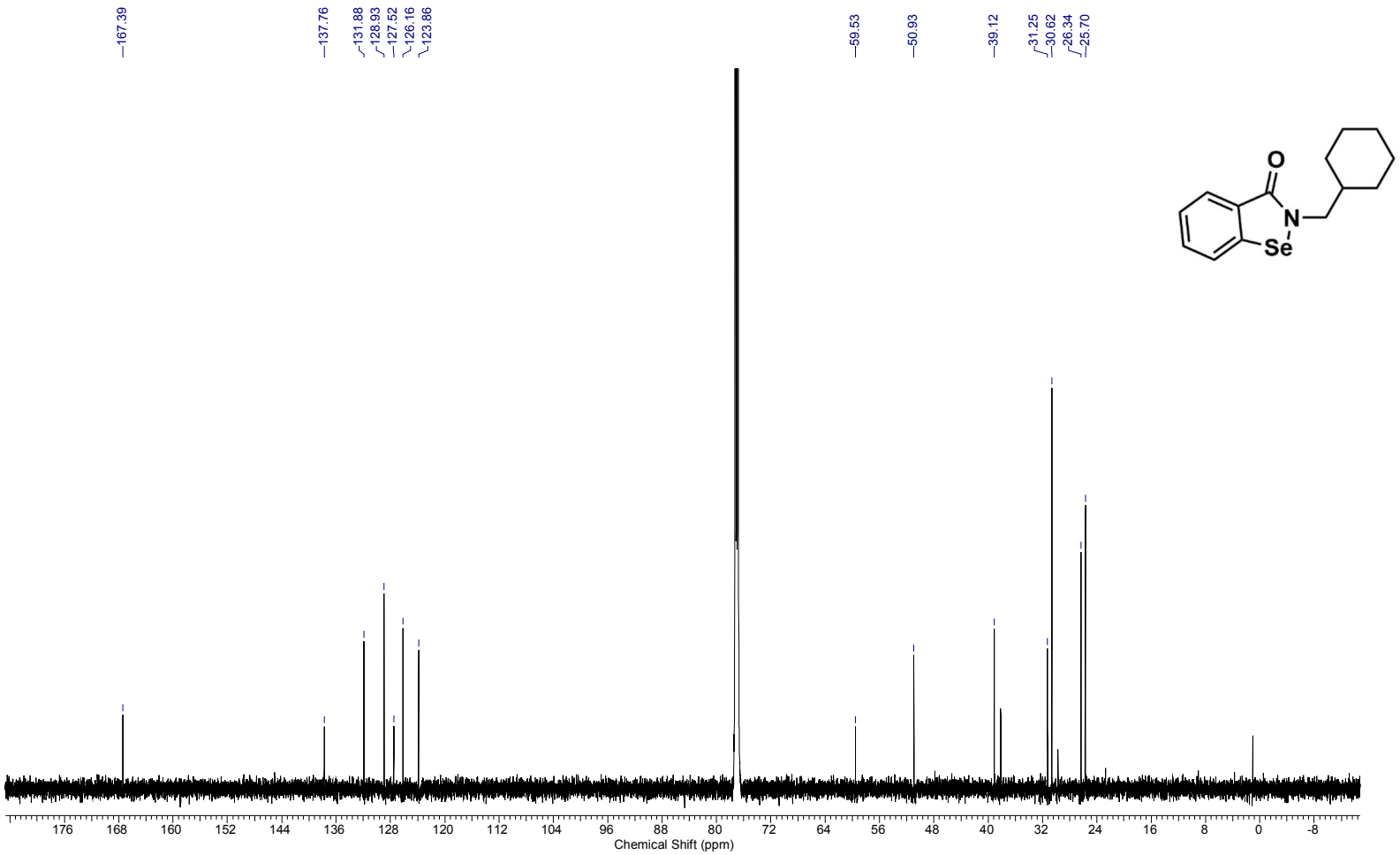
^{13}C-NMR of 2-allylbenzo[d][1,2]selenazol-3(2*H*)-one (1f)



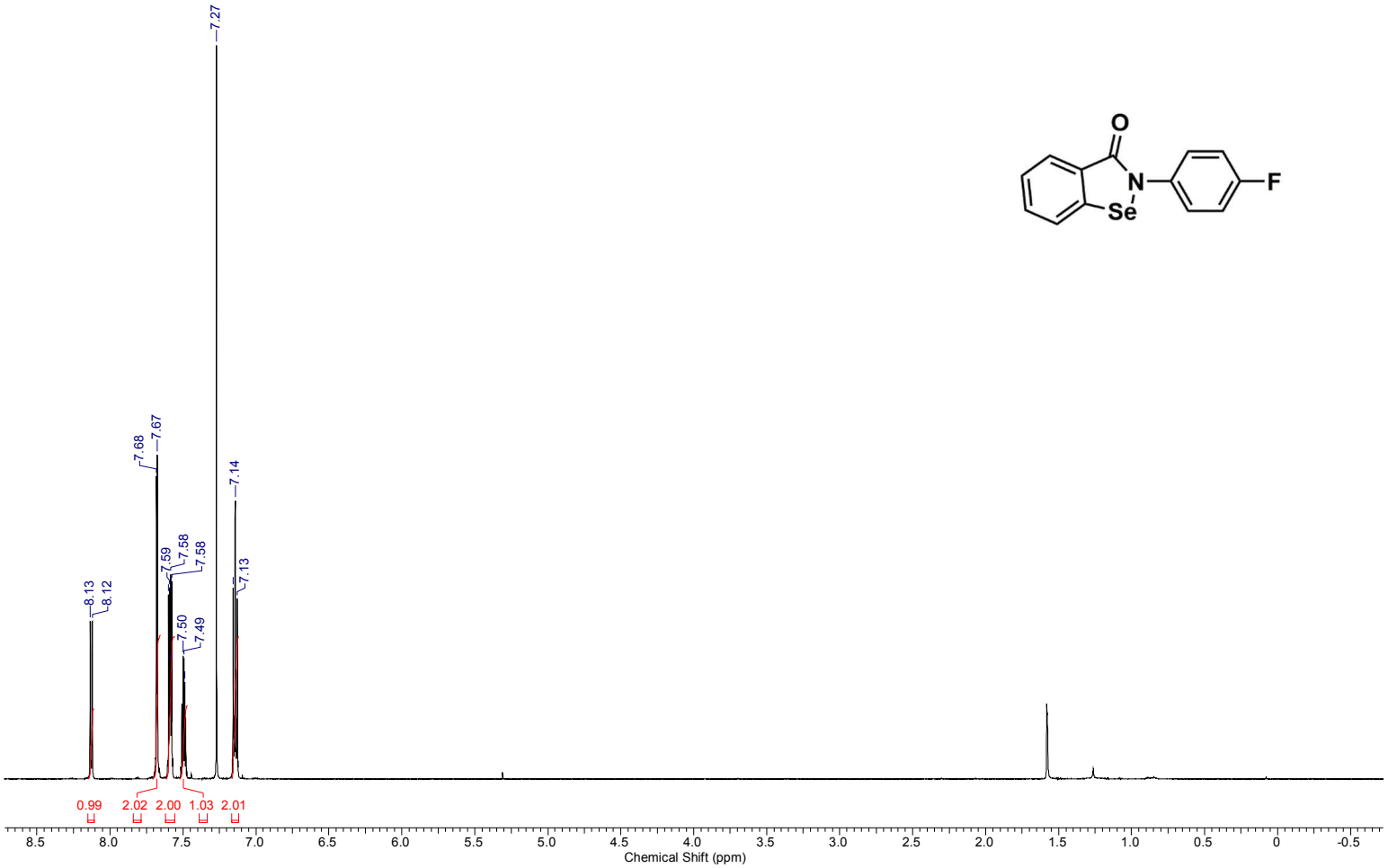
[1]H-NMR of 2-(cyclohexylmethyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1g)



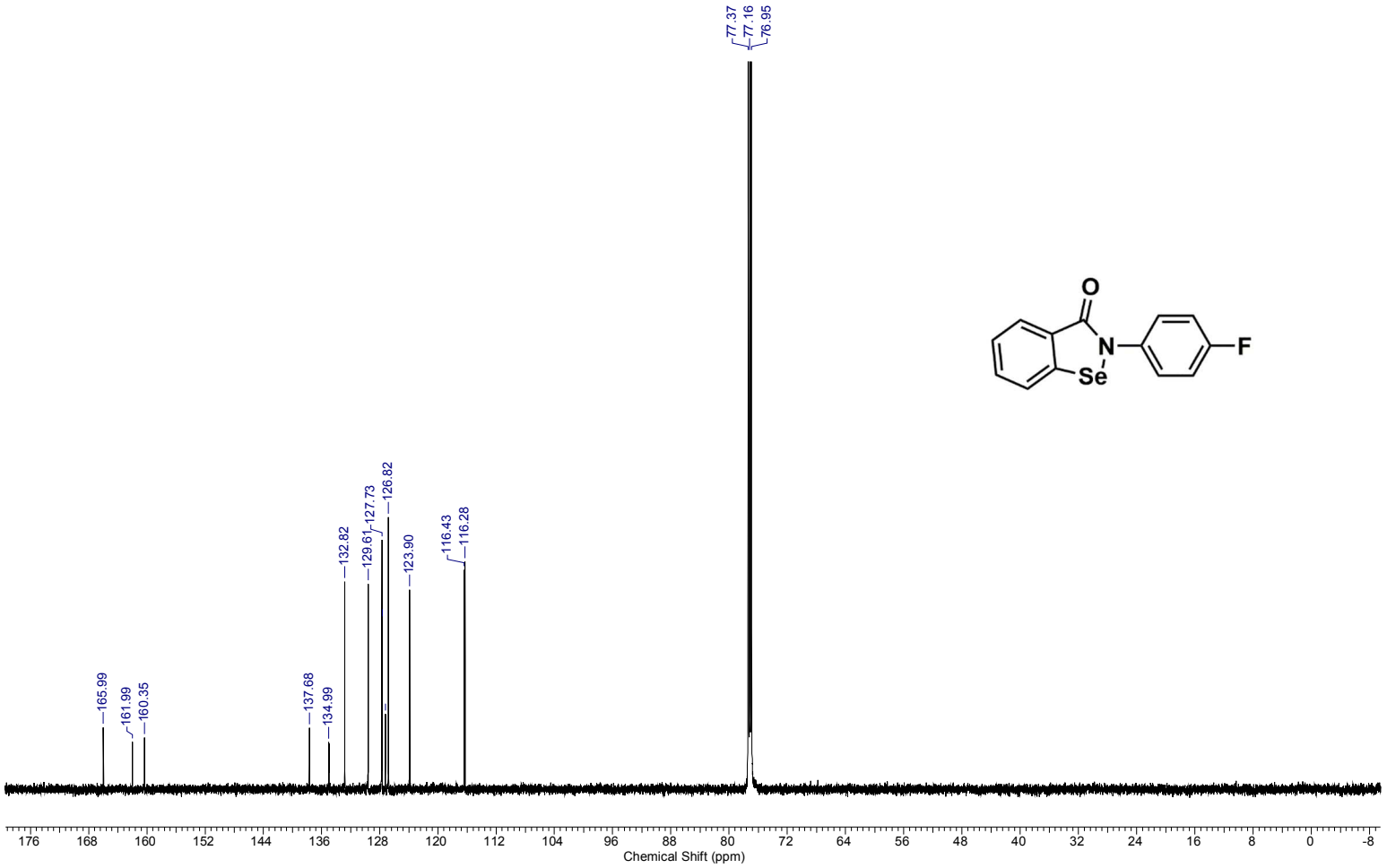
^{13}C-NMR of 2-(cyclohexylmethyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1g)



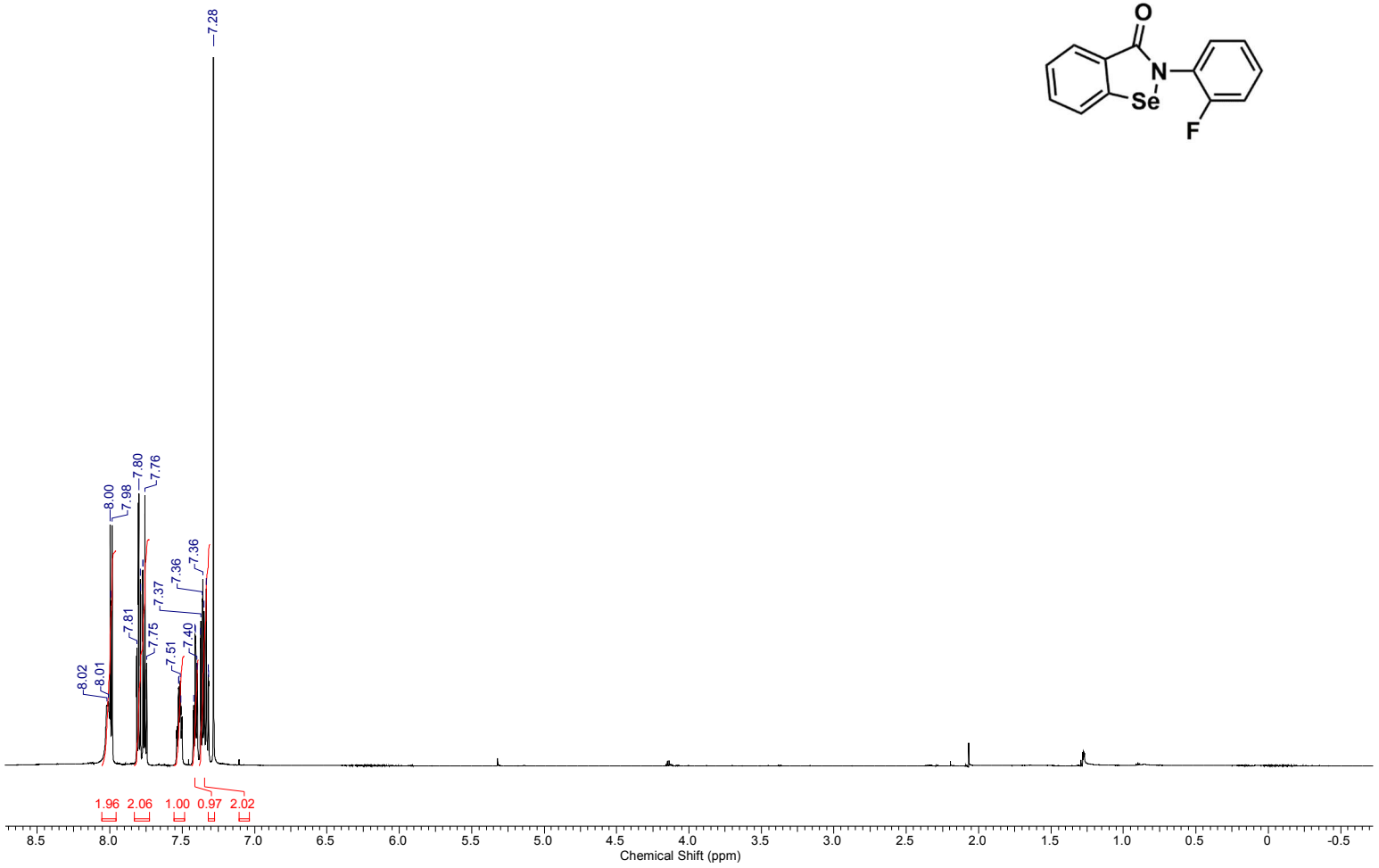
[1]H-NMR of 2-(4-fluorophenyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1h)



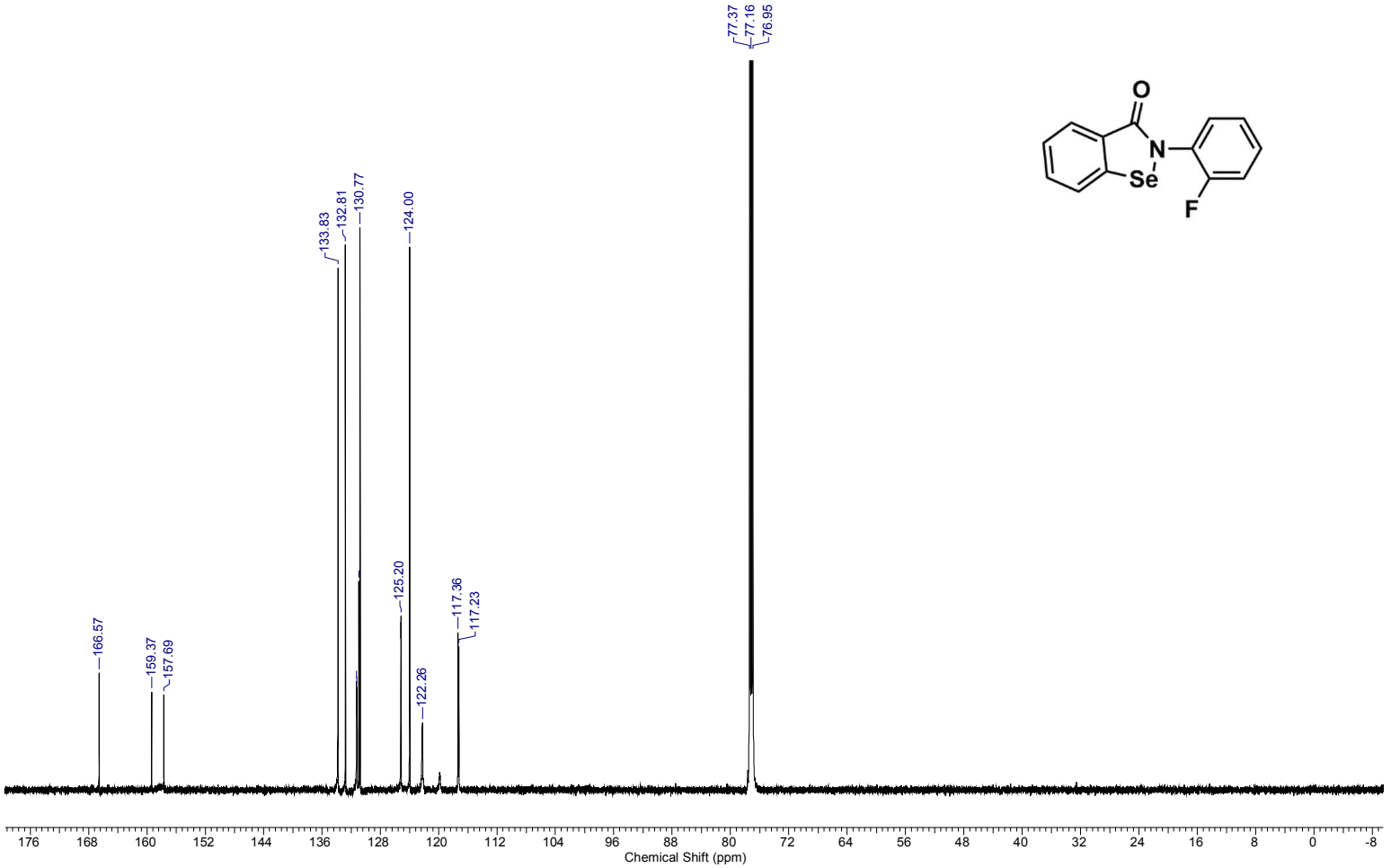
^{13}C-NMR of 2-(4-fluorophenyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1h)



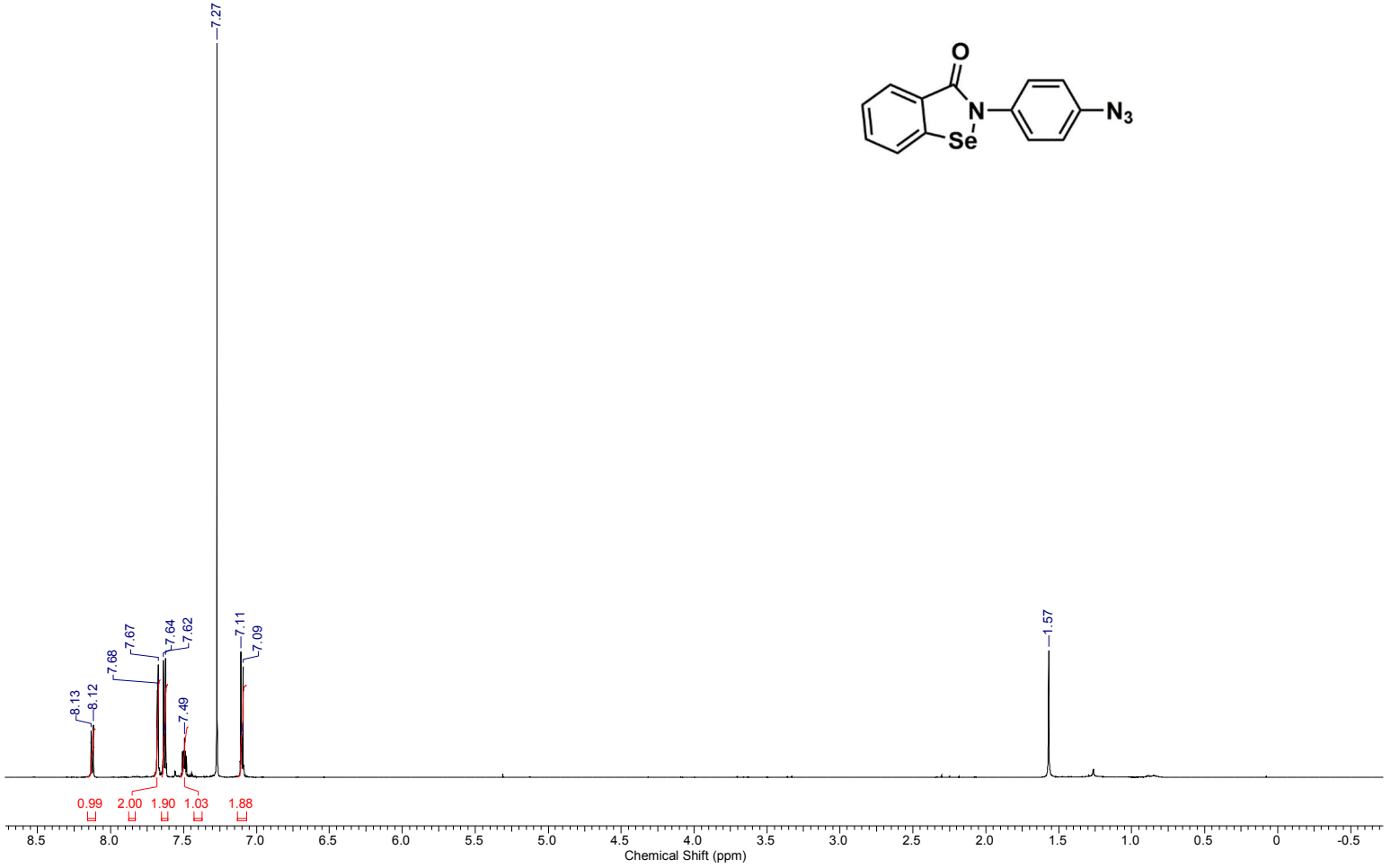
^{1}H-NMR of 2-(2-fluorophenyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1i)



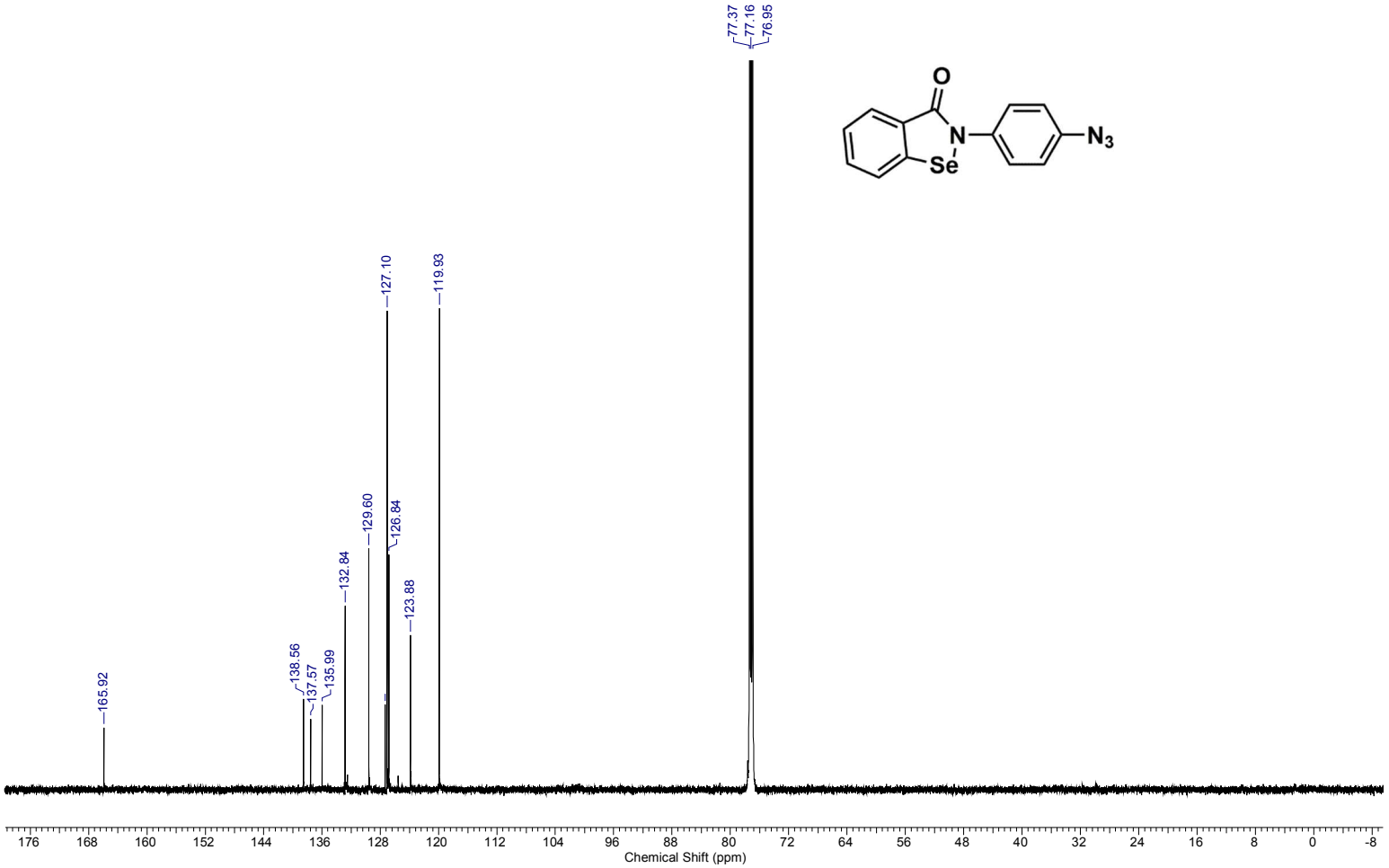
^{13}C-NMR of 2-(2-fluorophenyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1i)



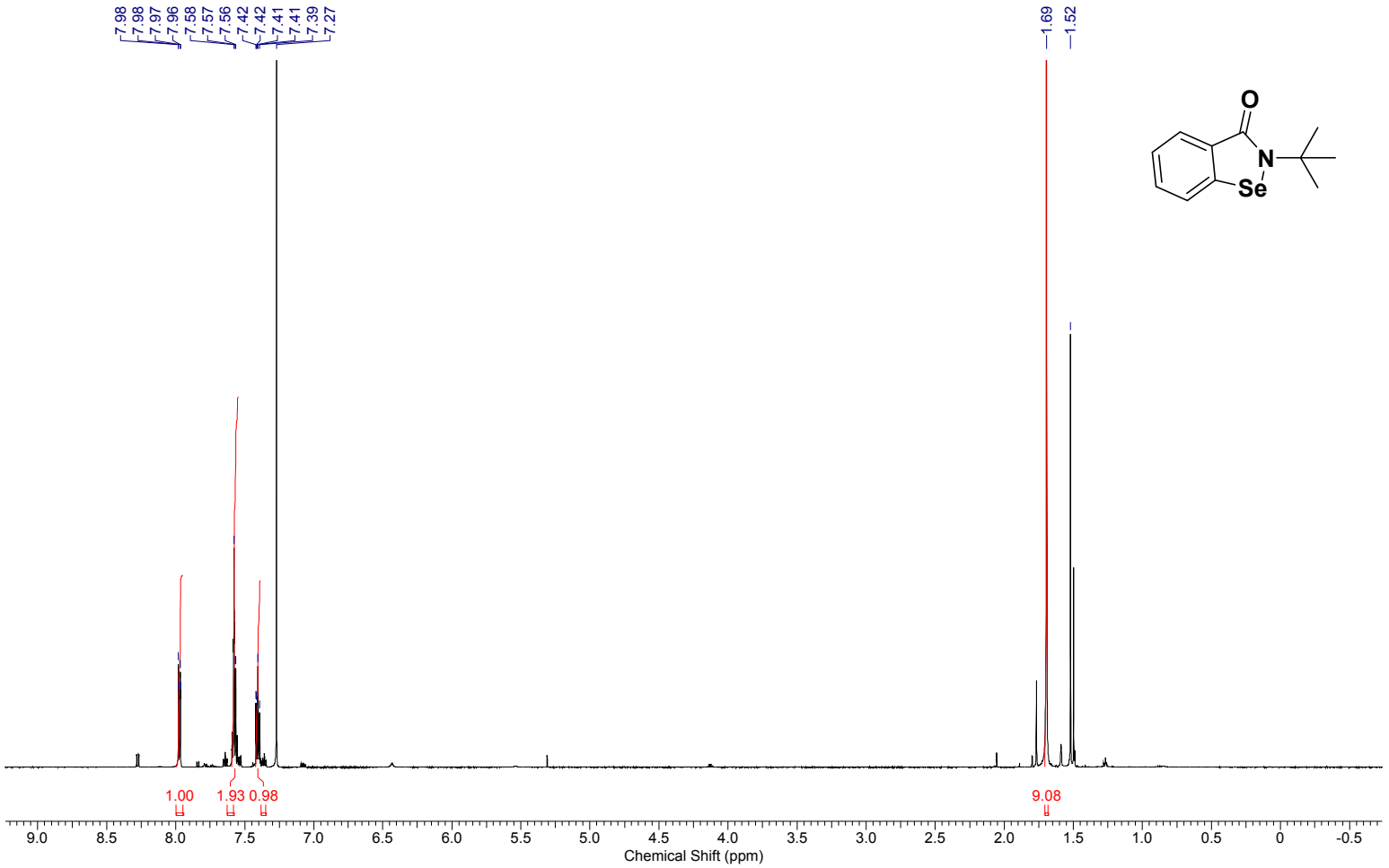
^{1}H-NMR of 2-(4-azidophenyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1j)



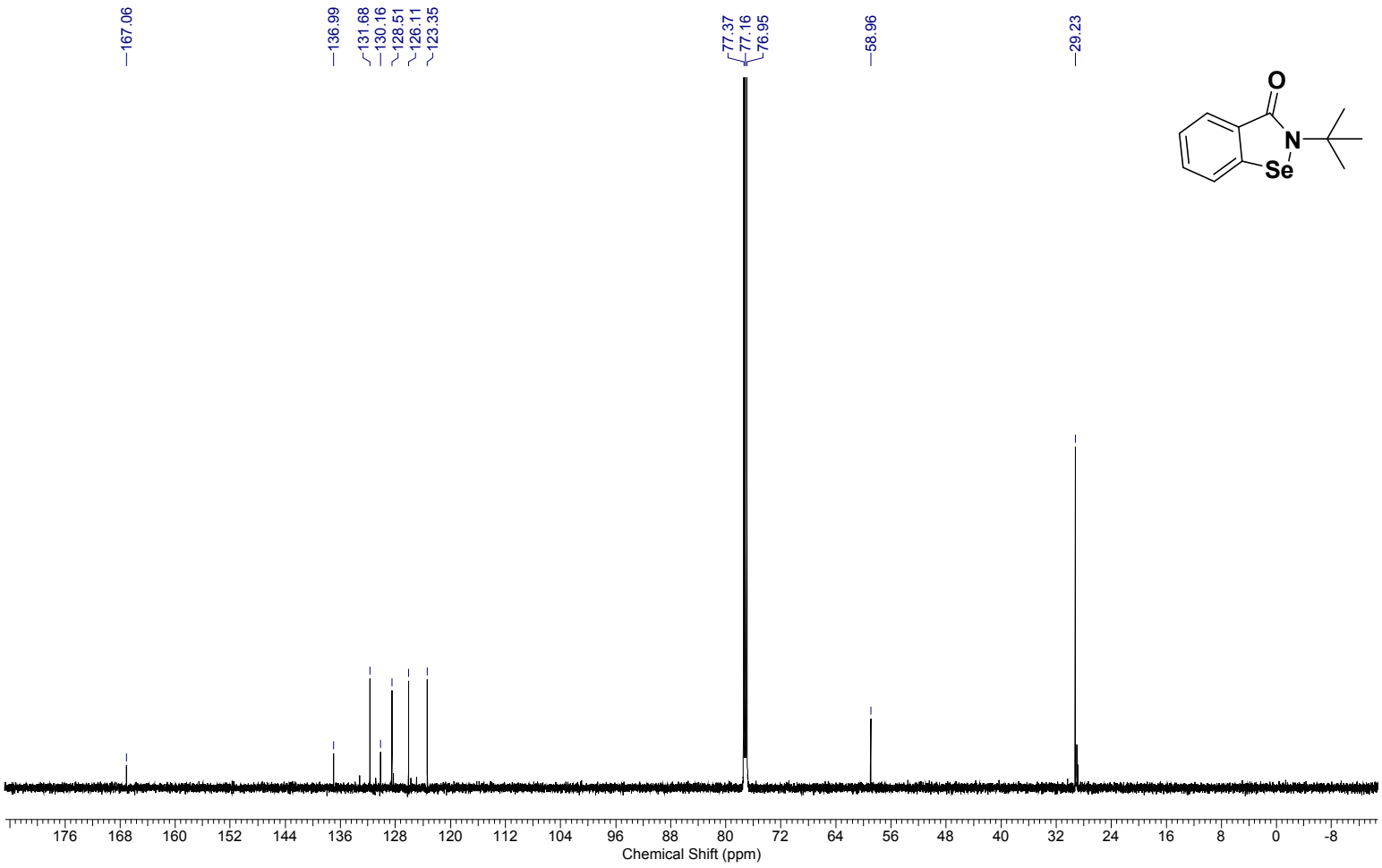
^{13}C-NMR of 2-(4-azidophenyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1j)



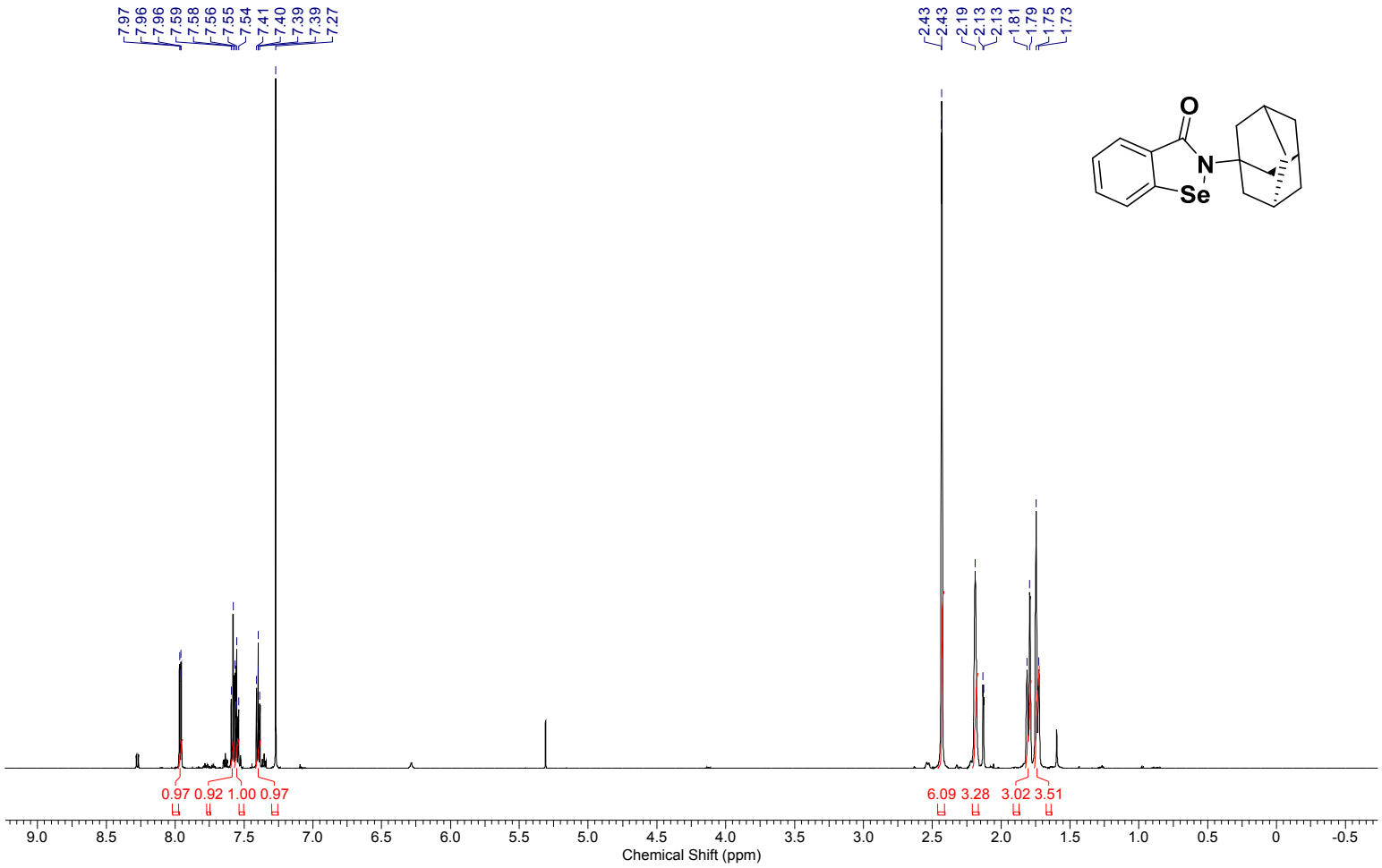
^{1}H-NMR of 2-(*tert*-butyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1k)



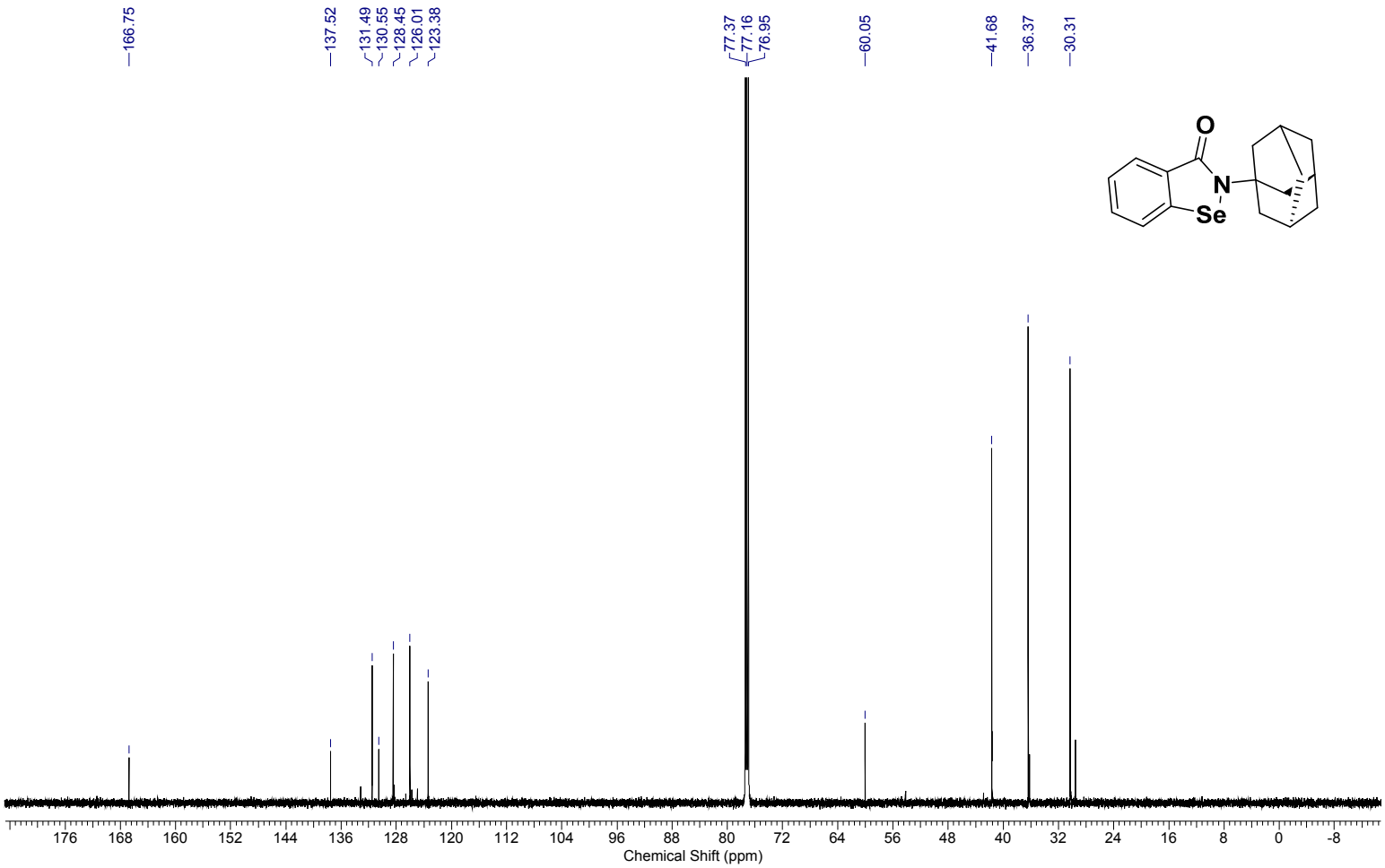
^{13}C-NMR of 2-(*tert*-butyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1k)



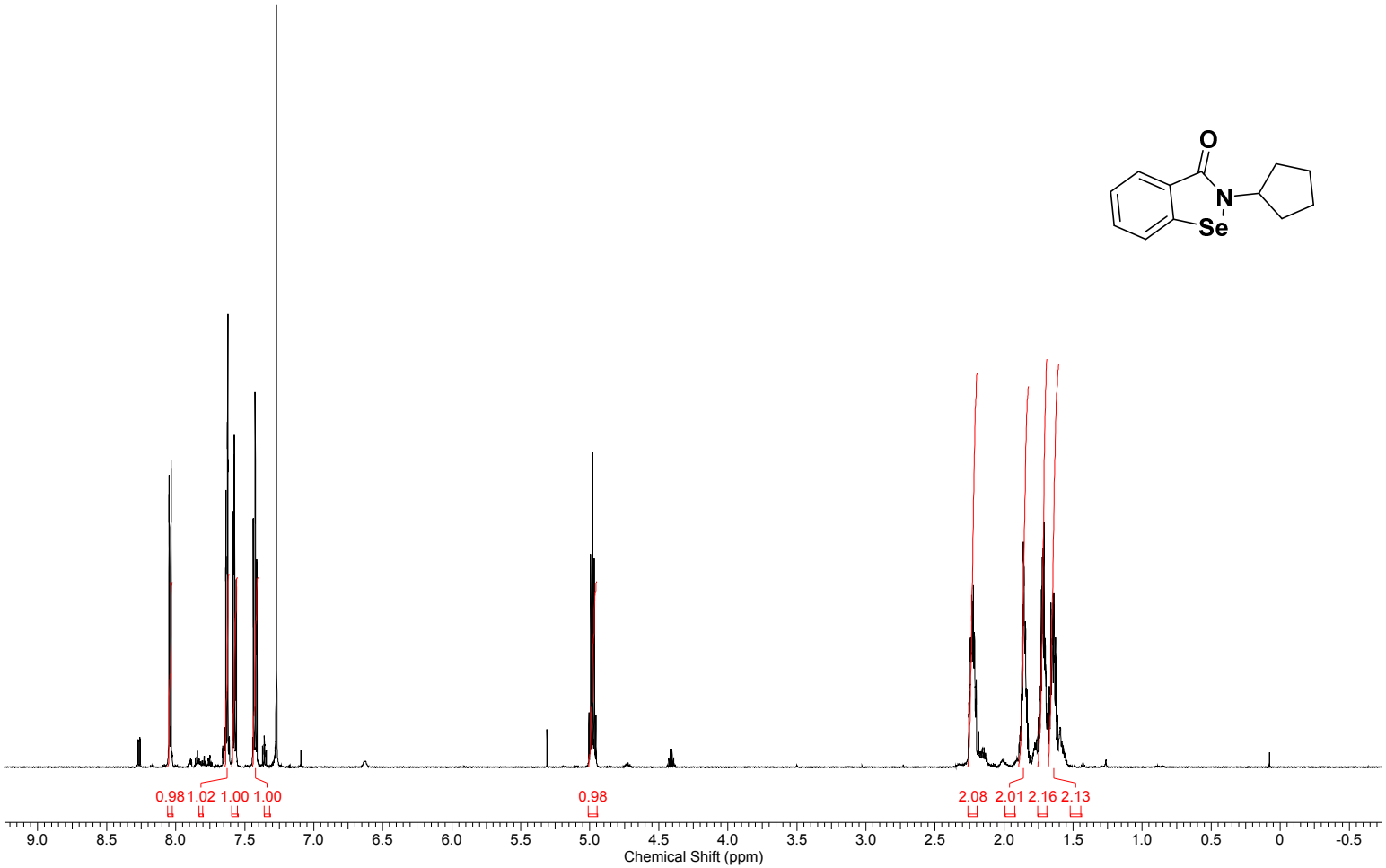
^{1}H-NMR of 2-((3*R*,5*S*)-adamantan-1-yl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1l)



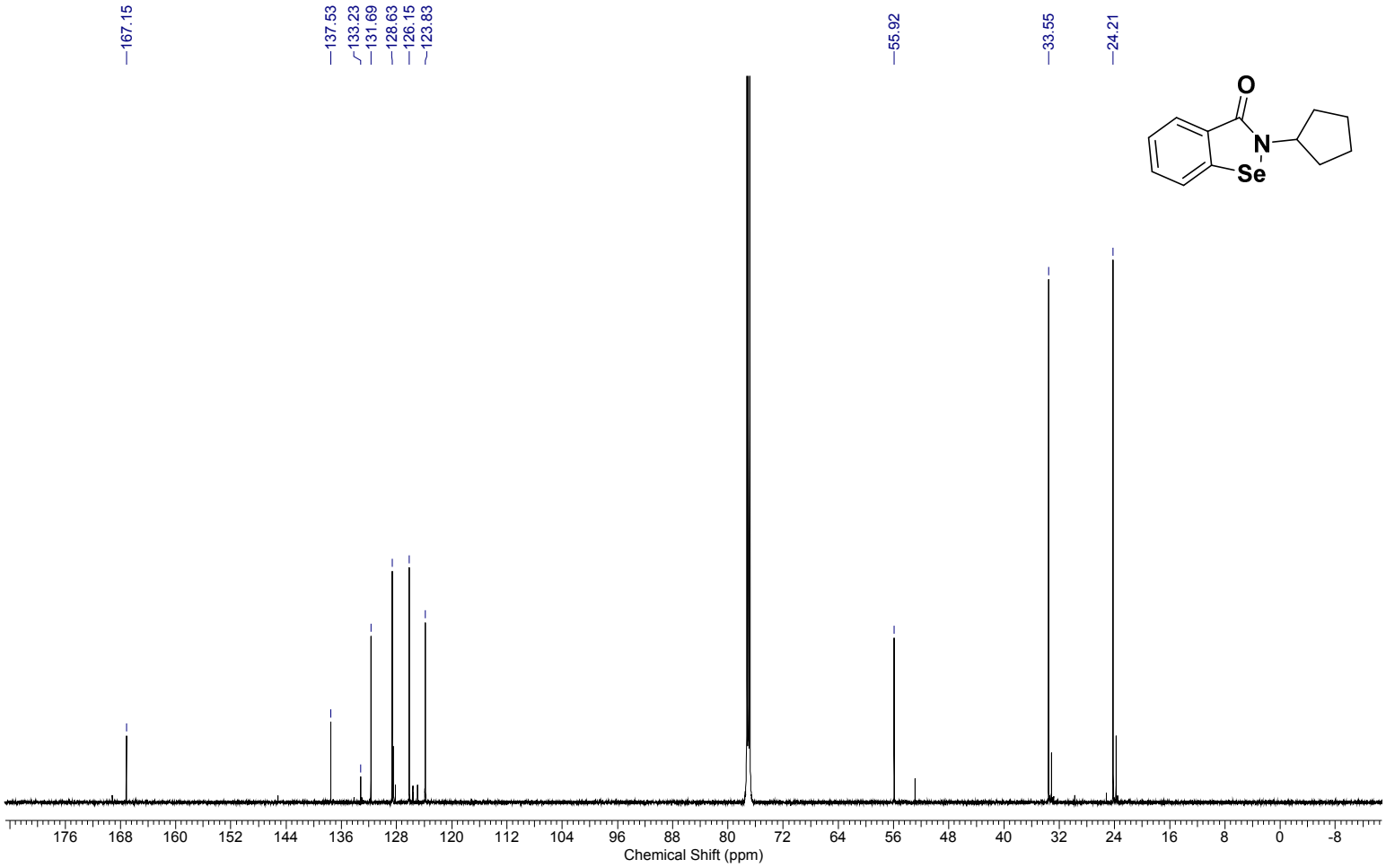
^{13}C-NMR of 2-((3*R*,5*S*)-adamantan-1-yl)benzo[*d*][1,2]selenazol-3(2*H*)-one (1l)



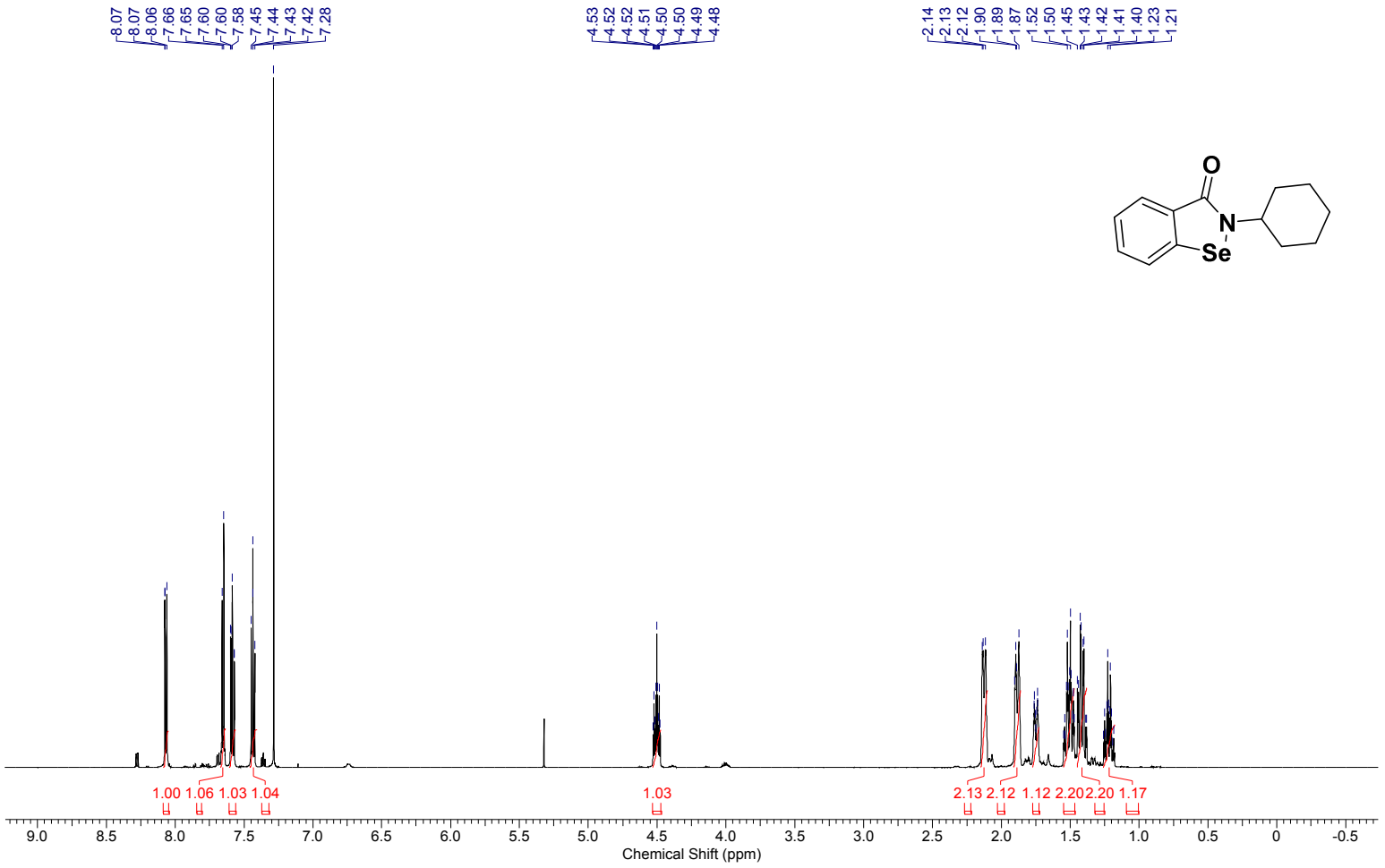
^{1}H-NMR of 2-cyclopentylbenzo[*d*][1,2]selenazol-3(2*H*)-one (1m)



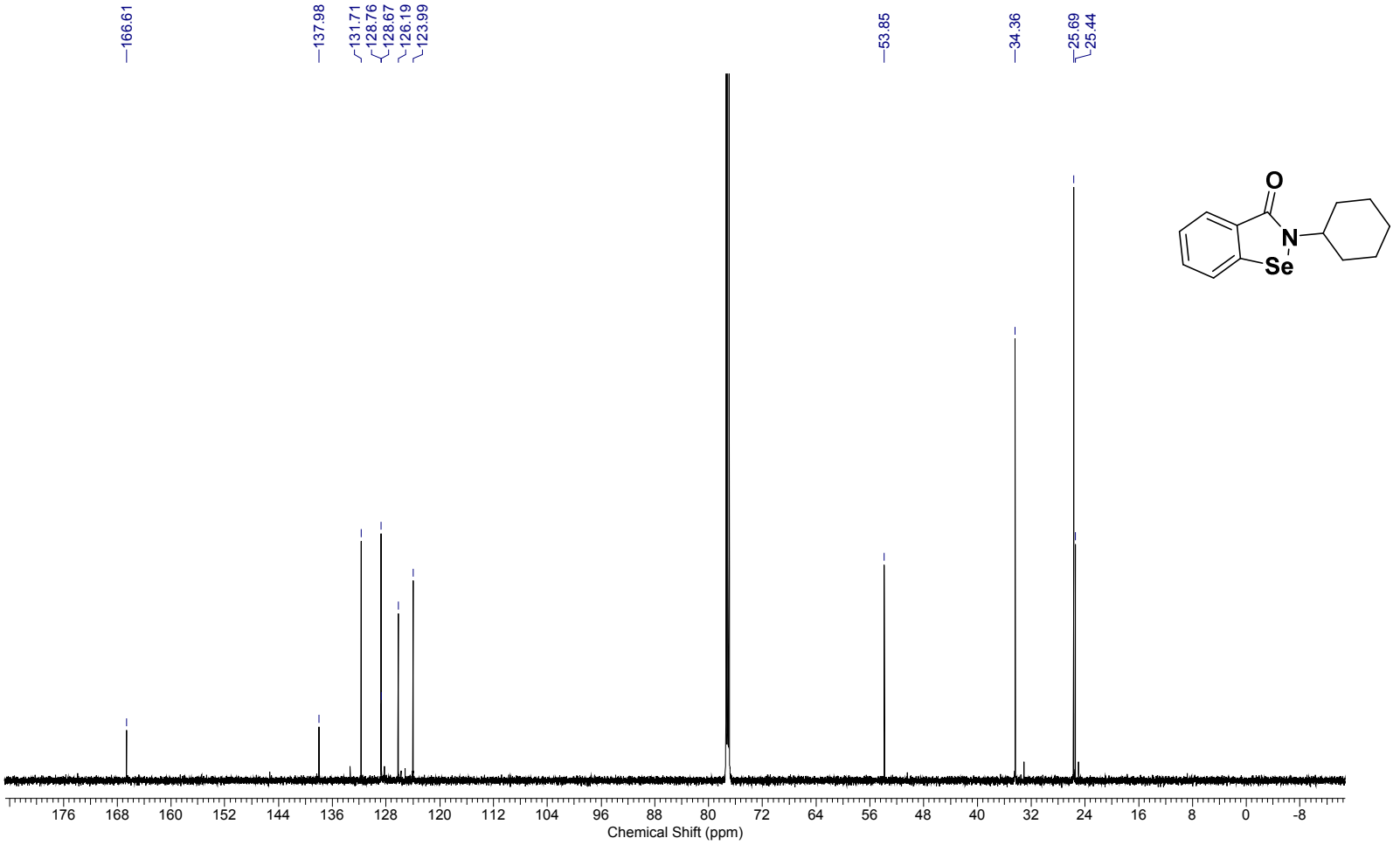
^{13}C-NMR of 2-cyclopentylbenzo[*d*][1,2]selenazol-3(2*H*)-one (1m)



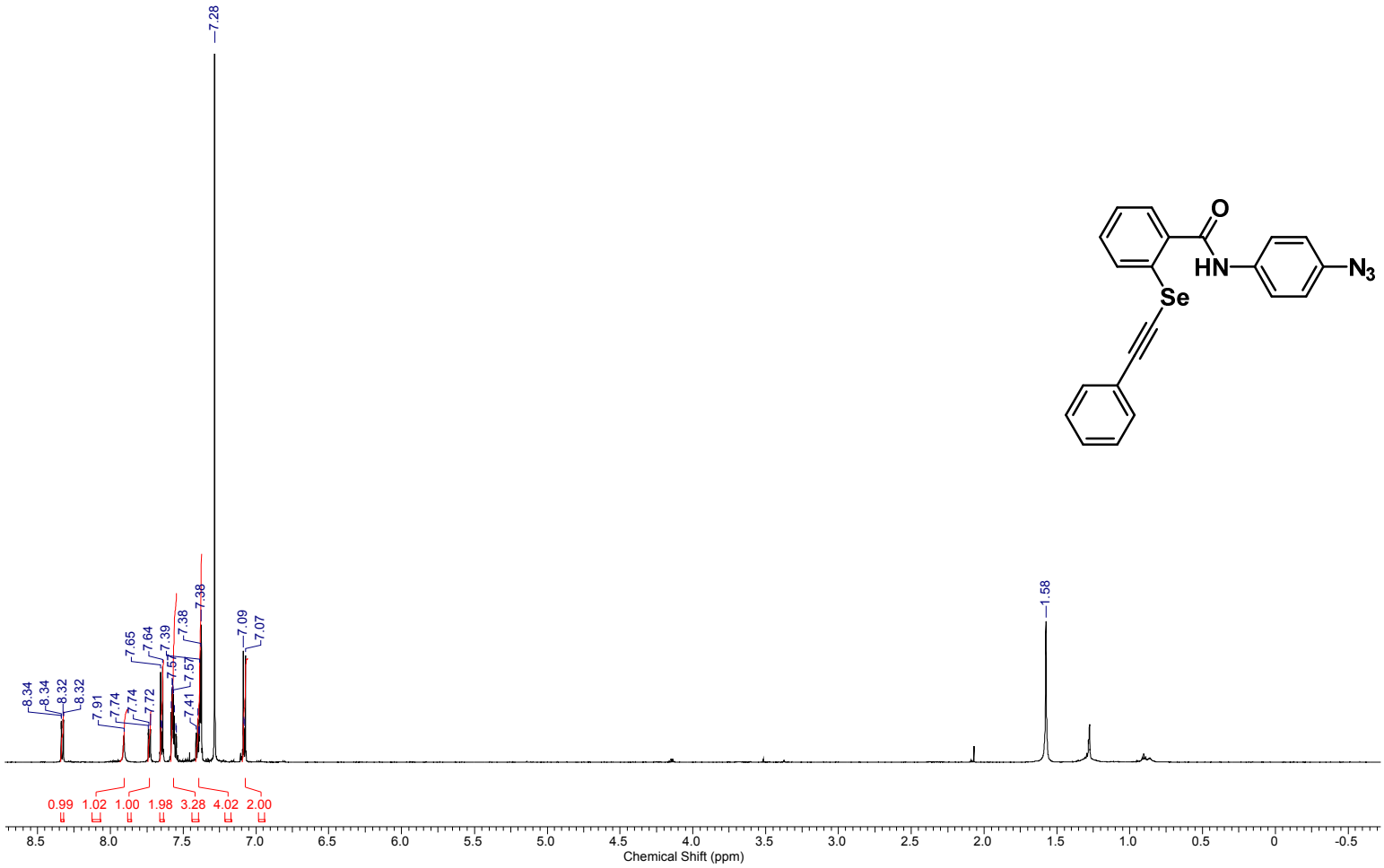
^{1}H-NMR of 2-cyclohexylbenzo[*d*][1,2]selenazol-3(2*H*)-one (1n)



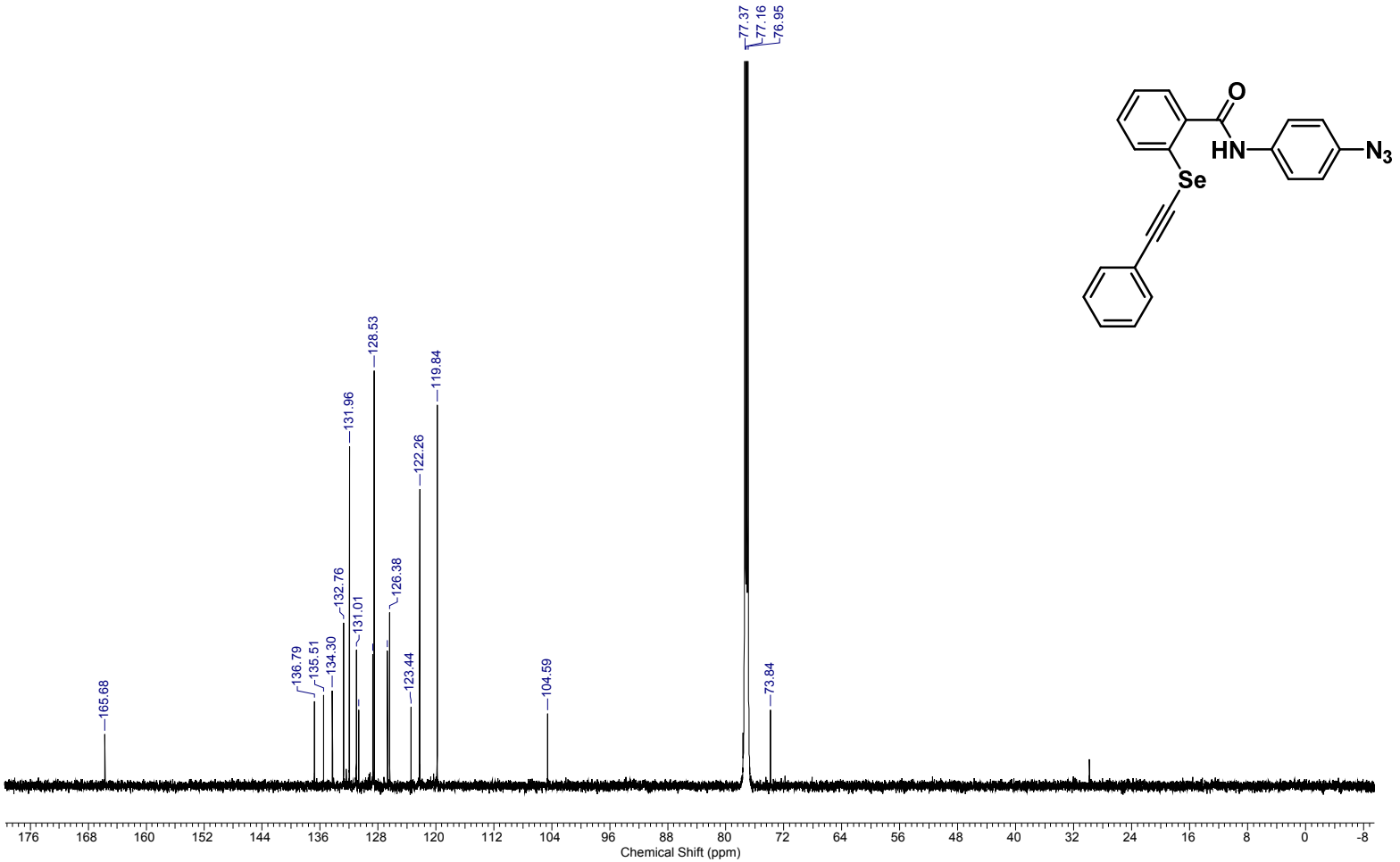
^{13}C-NMR of 2-cyclohexylbenzo[*d*][1,2]selenazol-3(2*H*)-one (1n)



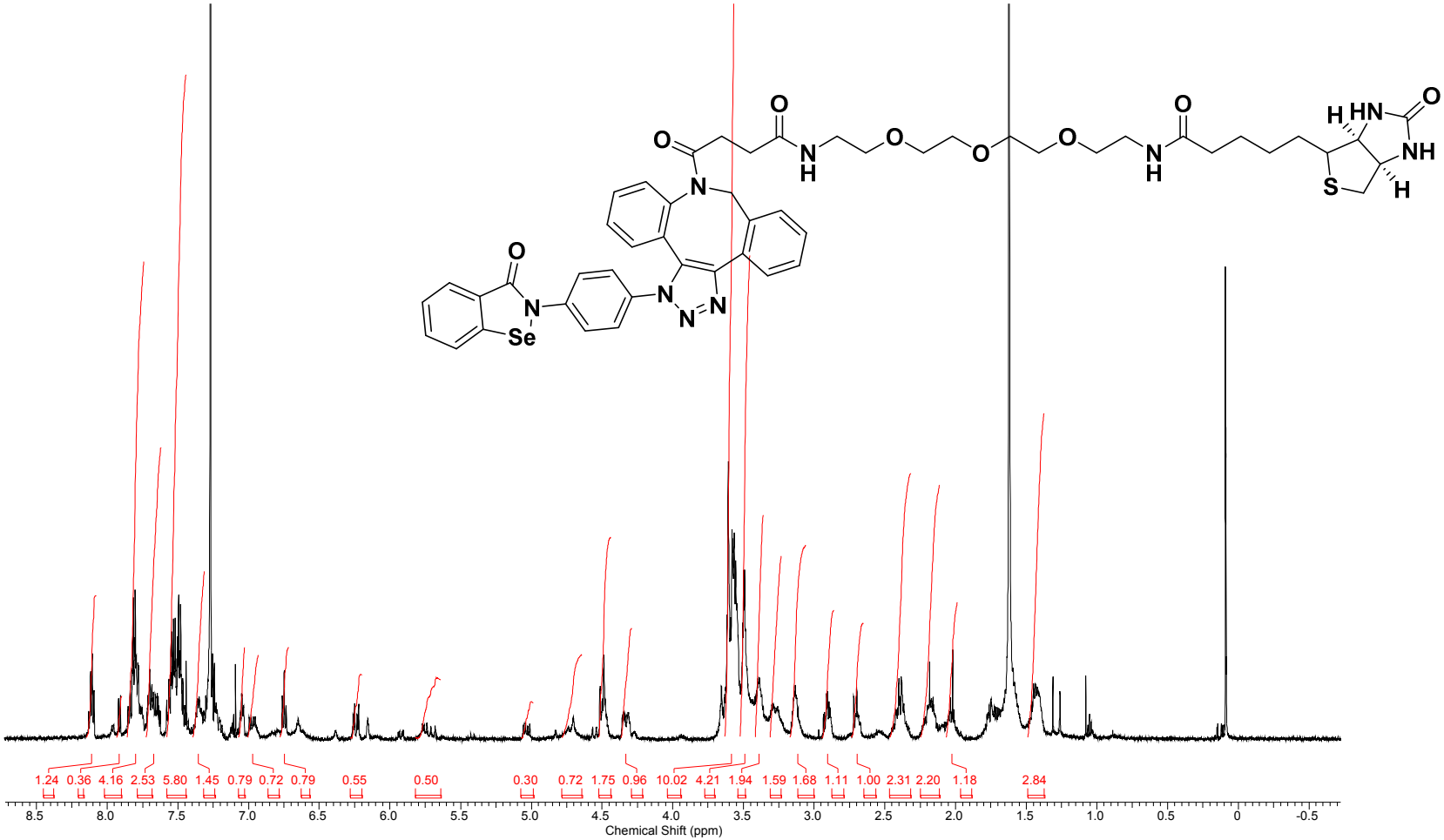
^{1}H-NMR of *N*-(4-azidophenyl)-2-((phenylethynyl)selanyl)benzamide (8)



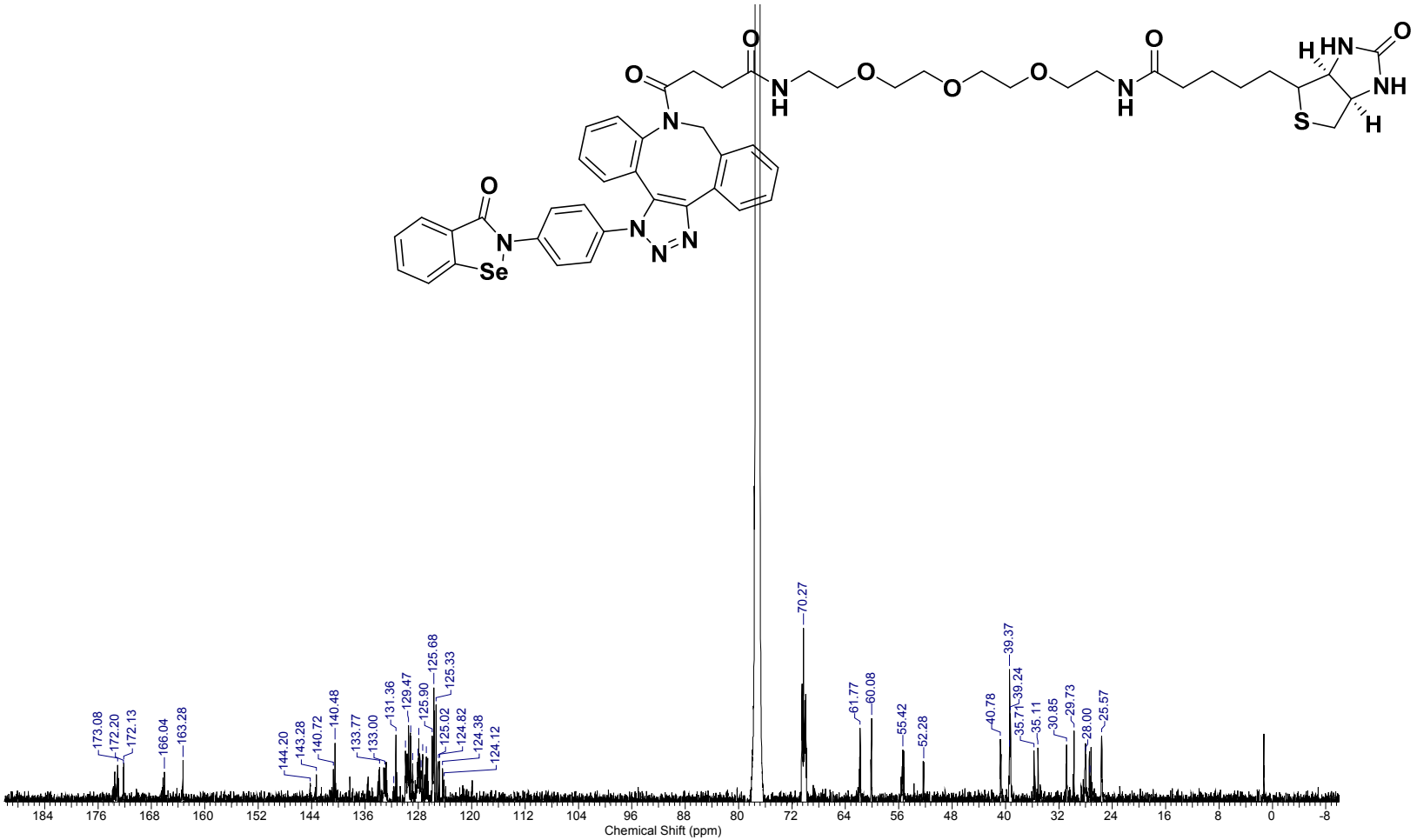
^{13}C-NMR of *N*-(4-azidophenyl)-2-((phenylethynyl)selanyl)benzamide (8)



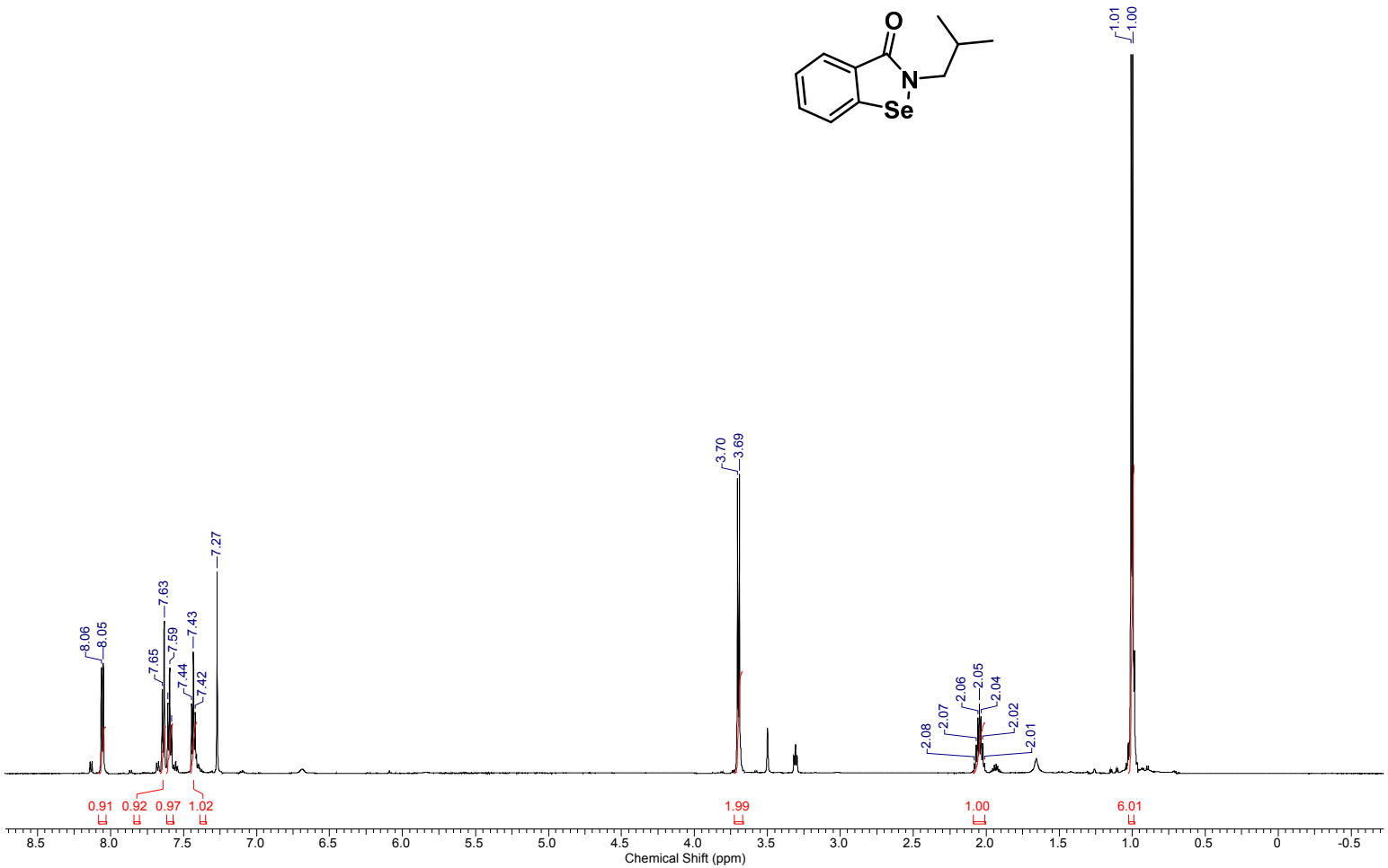
^{1}H-NMR of *N*-(13,16-dioxo-16-(3-(4-(3-oxobenzo[*d*][1,2]selenazol-2(3*H*)-yl)phenyl)-3,9-dihydro-8*H*-dibenzo[*b*,*f*][1,2,3]triazolo[4,5-*d*]azocin-8-yl)-3,6,9-trioxa-12-azahexadecyl)-5-((3a*S*,6a*R*)-2-oxohexahydro-1H-thieno[3,4-*d*]imidazol-4-yl)pentanamide (10)



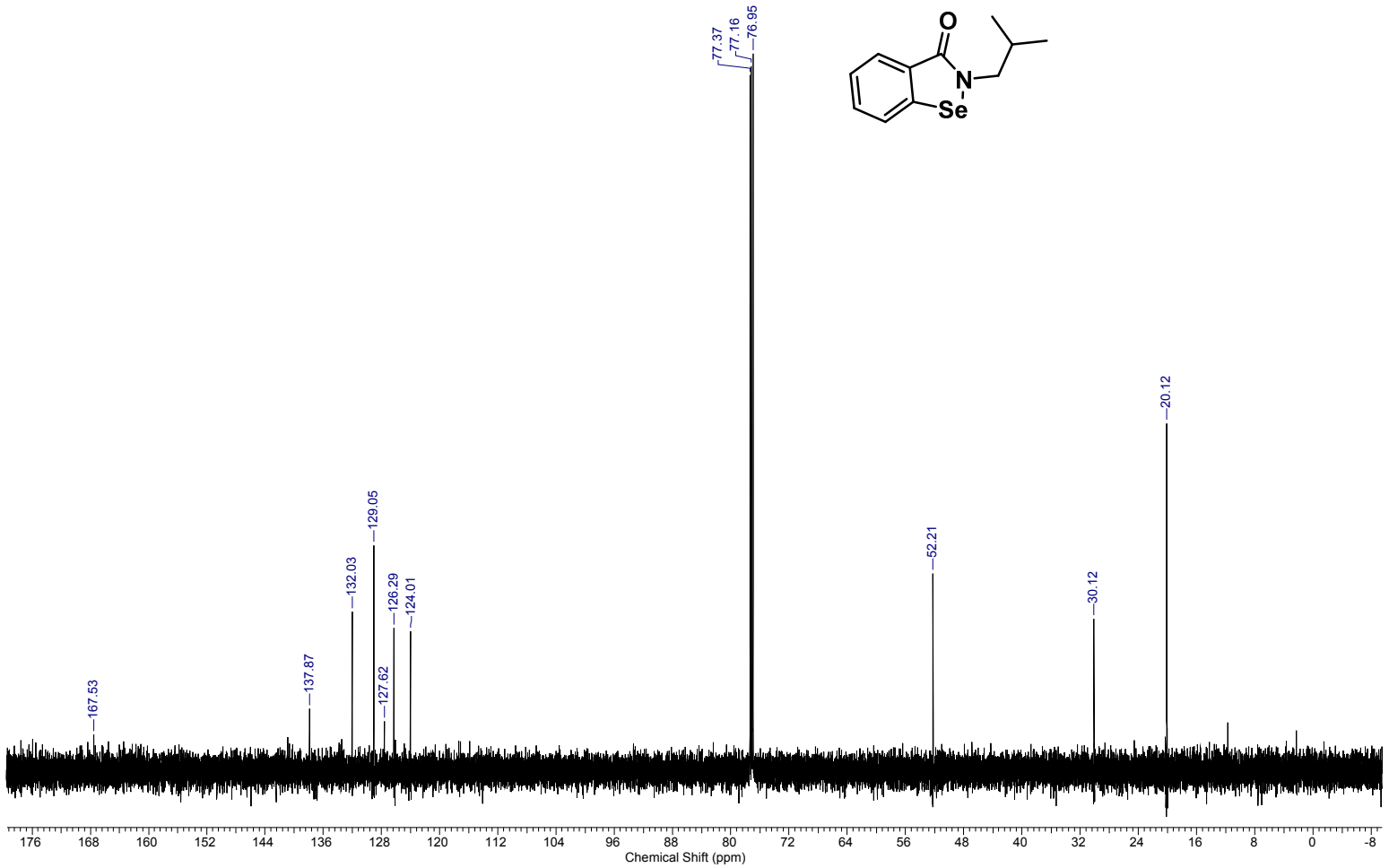
^{13}C-NMR of *N*-(13,16-dioxo-16-(3-(4-(3-oxobenzo[*d*][1,2]selenazol-2(3*H*)-yl)phenyl)-3,9-dihydro-8*H*-dibenzo[*b*,*f*][1,2,3]triazolo[4,5-*d*]azocin-8-yl)-3,6,9-trioxa-12-azahexadecyl)-5-((3a*S*,6a*R*)-2-oxohexahydro-1H-thieno[3,4-*d*]imidazol-4-yl)pentanamide (10)



^{1}H-NMR of 2-isobutylbenzo[*d*][1,2]selenazol-3(2*H*)-one (1o)



^{13}C-NMR of 2-isobutylbenzo[*d*][1,2]selenazol-3(2*H*)-one (1o)



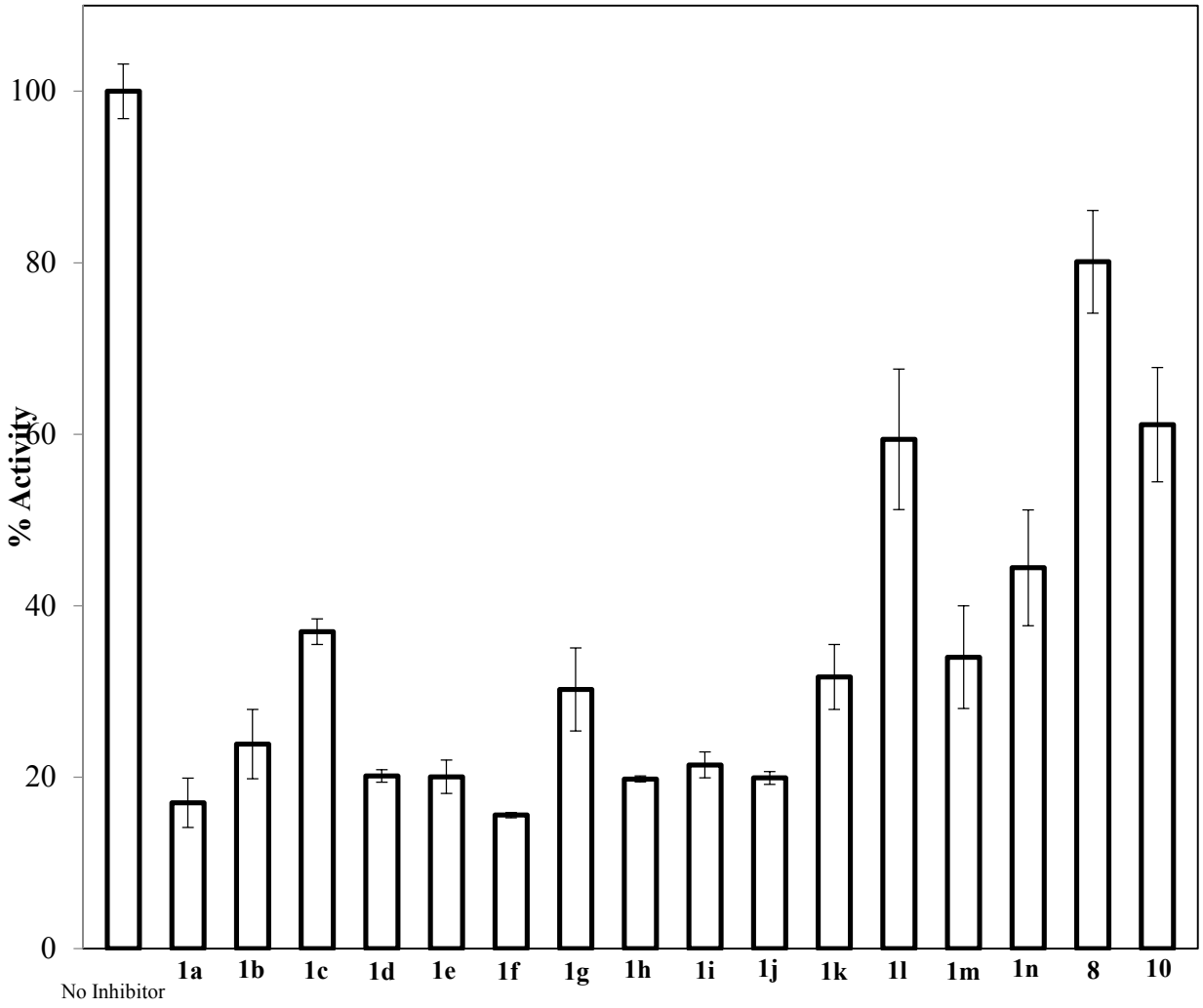


Figure S7. The enzymatic activity of *Mtb* Ag85C was evaluated after 40 min of incubation with 5 µM 2-alkyl-1,2-benzisoselenazol-3(2*H*)-one **1a-1n**, **8**, and **10**. Enzyme activity was normalized to the control reaction that contained no inhibitor. The error bars are calculated by performing each experiment in triplicate.

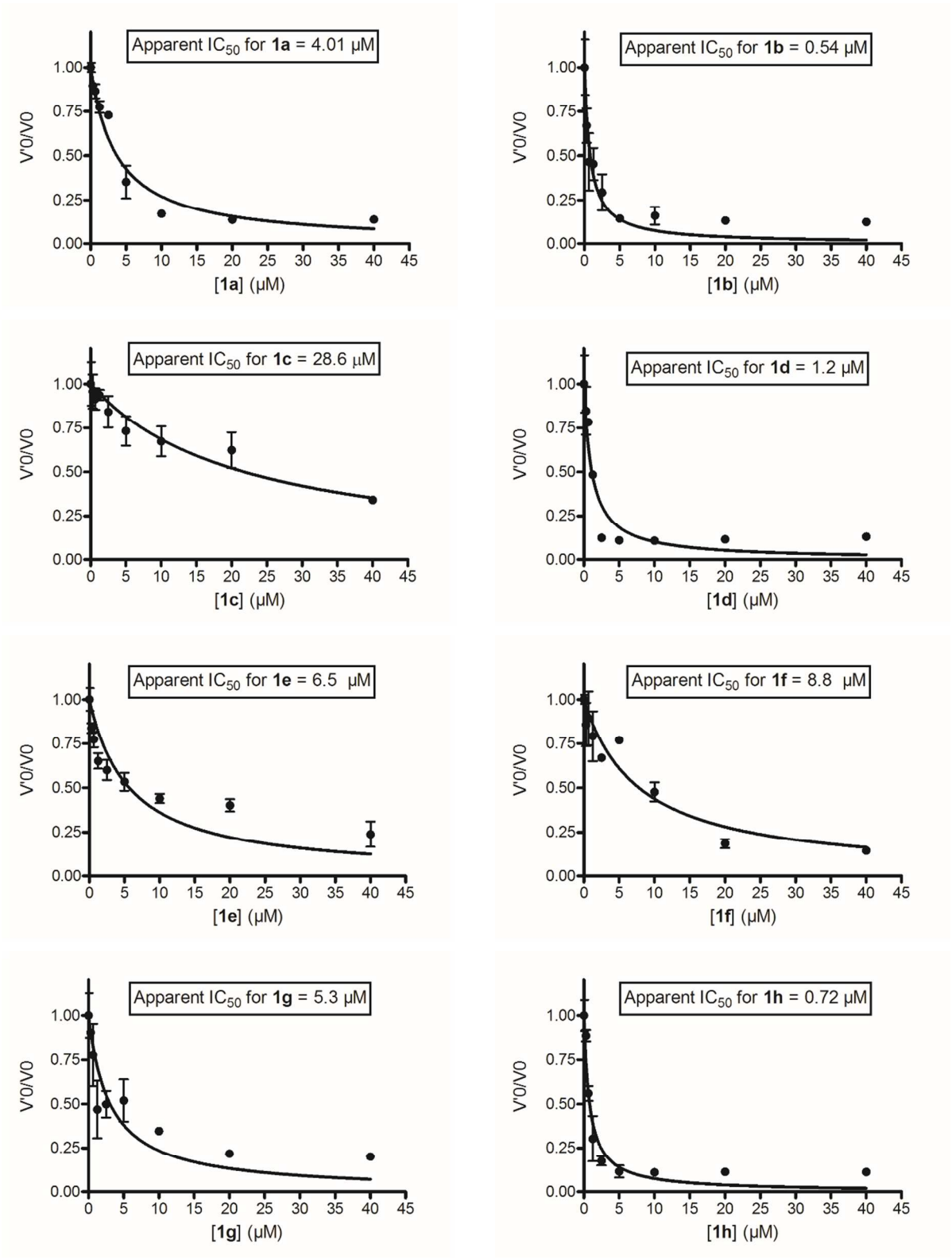


Figure S8A. Apparent IC50 for 1,2-benzisoselenazol-3(2*H*)-ones. Error bars are calculated by performing each experiment in triplicate.

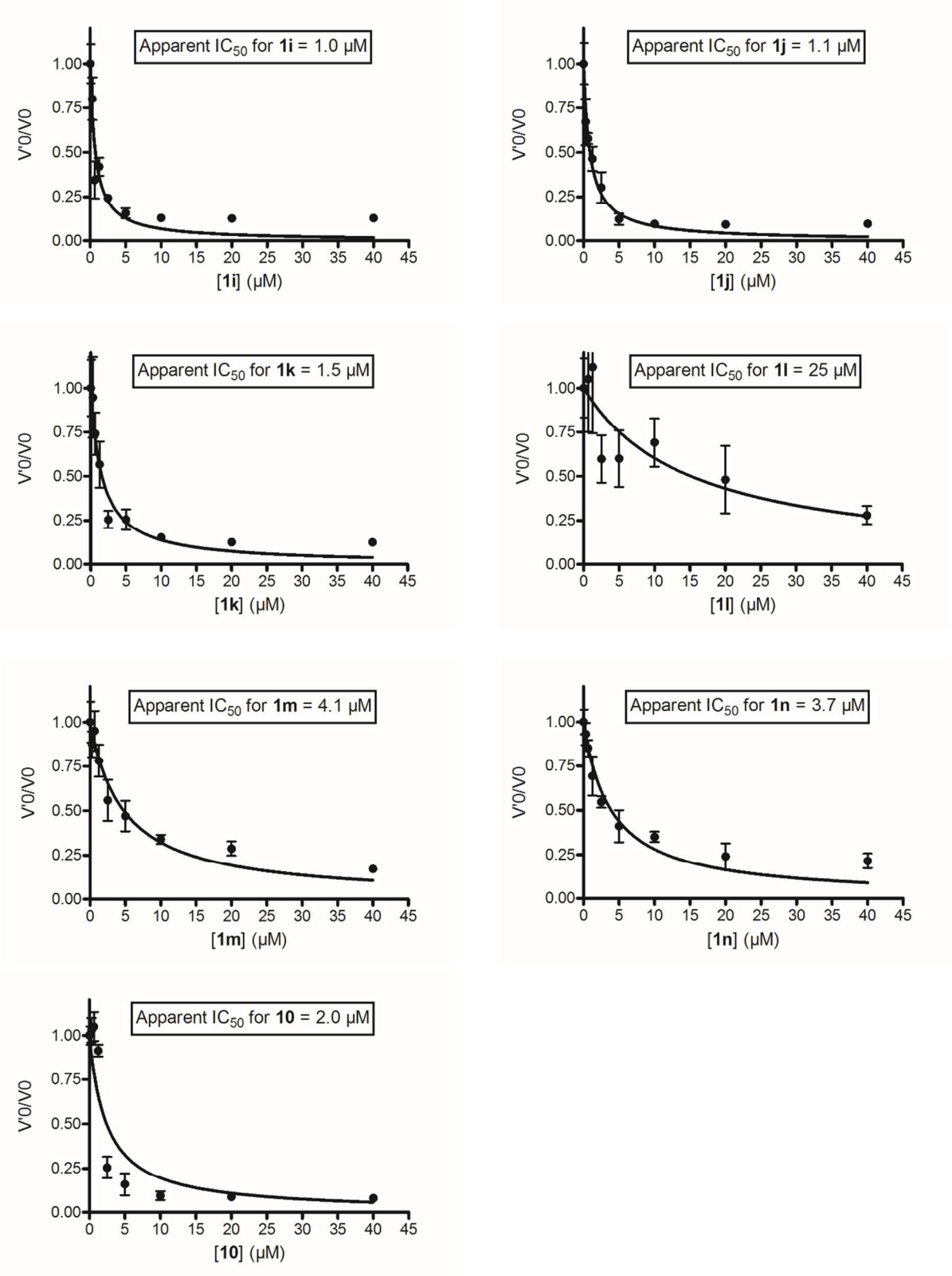


Figure S8B. Apparent IC50 for 2-alkyl-1,2-benzisoselenazol-3(2*H*)-ones. Error bars are calculated by performing each experiment in triplicate.