Supporting Information

New UV-light initiated intramolecular Se-N bond formation

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II. Synthesis of diselenides **21a** and **22a**

To a solution of benzisoselenazol-3(2H)-one (1.0 mmol; obtained by our previously published procedure (Method A): A.J Pacuła, K. B. Kaczor, A. Wojtowicz, J. Antosiewicz, A. Janecka, A. Długosz, T. Janecki and J. Scianowski, *Bioorg. Med. Chem*., 2017, **25**, 126–131) in methanol (10 ml) cooled to 0°C, sodium borohydride (1.0 mmol) was added and the mixture was stirred for 1h. Water (15ml) was added and the mixture was oxidized with air for 1h. Formed precipitate was filtered and dried in air.

2,2'-Diselenobis((4-(Trifluoromethyl)phenyl)benzamide) 21a

Yield: 70%, mp 228-230°C;
^{1}H NMR (700 MHz, DMSO) δ = 7.42 (dt, *J*=7.0, 0.7 Hz, 1H$_{ar}$), 7.47 (dt, *J*=7.0, 1.4 Hz, 1H$_{ar}$), 7.75 (d, *J*= 8.4, 2H$_{ar}$), 7.78 (dd, *J*=7.7, 0.7 Hz, 1H$_{ar}$), 7.97-7.99 (m, 3H$_{ar}$), 10.86 (s, NH) ppm; ^{13}C NMR (100.61 Hz, DMSO) δ = 120.24, 120.89, 123.47, 124.40, 124.72, 126.17, 126.48 (q), 127.03, 129.40, 130.82, 132.13, 132.63, 132.88, 133.85, 139.08, 142.80, 167.26 (C=O) ppm; ^{77}Se (133.55 MHz, DMSO), δ = 446.68 ppm; IR: 3314, 1649, 1615, 1600, 1563, 1523, 1517, 1461, 1429, 1409, 1322, 1269, 1256, 1186, 1157, 1114, 1067, 1047, 1017 cm^{-1}; Elemental Anal. Calcd for $C_{28}H_{18}F_6N_2O_2Se_2$ (687.96): C, 49.00; H, 2.64, Found: C, 48.89; H, 2.59. Elemental Anal. Calcd for $C_{28}H_{18}F_6N_2O_2Se_2$ (687.96): C, 49.00; H, 2.64, Found: C, 49.23; H, 2.71.

2,2'-Diselenobis((2-(Trifluoromethyl)phenyl)benzamide) 22a

Yield: 74%, mp 277-279°C;
^{1}H NMR (700 MHz, DMSO) δ = 7.45 (dt, *J*=7.7, 1.4 Hz, 1H$_{ar}$), 7.49 (dt, *J*=8.4, 1.4 Hz, 1H$_{ar}$), 7.59-7.61 (m, 2H$_{ar}$), 7.78-7.81 (m, 2H$_{ar}$), 7.85 (d, *J*= 7.7, 1H$_{ar}$), 8.00 (d, *J*= 7.0, 1H$_{ar}$), 10.51 (s, NH) ppm; ^{13}C NMR (100.61 Hz, DMSO) δ = 122.71, 125.43, 126.95, 127.13 (q), 128.33, 129.02, 130.66, 131.81, 132.75, 133.01, 133.80, 135.72, 167.89 (C=O) ppm; ^{77}Se (133.67MHz, DMSO), δ = 446.29 ppm; IR: 3280, 1643, 1610, 1587, 1525, 1487, 1454, 1321, 1306, 1288, 1267, 1256, 1173, 1141, 1110, 1060, 1036 cm^{-1}; Elemental Anal. Calcd for $C_{28}H_{18}F_6N_2O_2Se_2$ (687.96): C, 49.00; H, 2.64, Found: C, 48.89; H, 2.59

II. Synthesis of benzisoselenazol-2(H)-ones **21** and **22**

Method A: To a solution of amine (2.0 mmol) and triethylamine (4.0 mmol) in dichloromethane (10 ml) 2-(chloroseleno)benzoyl chloride (2.0 mmol) was added. The mixture was stirred for 24h at room temperature, poured on water and extracted with DCM. The combined organic layers were dried over anhydrous magnesium sulfate and evaporated. The crude product was purified by column chromatography (silica gel, dichlorometane).

Method B: A solution of the starting diselenide **21a**/**22a** in acetonitrile (0.01M) was placed in a 5ml quartz cuvette and irradiated for 1h by 250 nm wavelength UV lamp. The solution was poured into a 10ml flask and evaporated. The final product was isolated by column chromatography (DCM, neutral aluminium oxide).

***N*-4-(Trifluoromethyl)phenyl-1,2-benzisoselenazol-3(2*H*)-one 21**

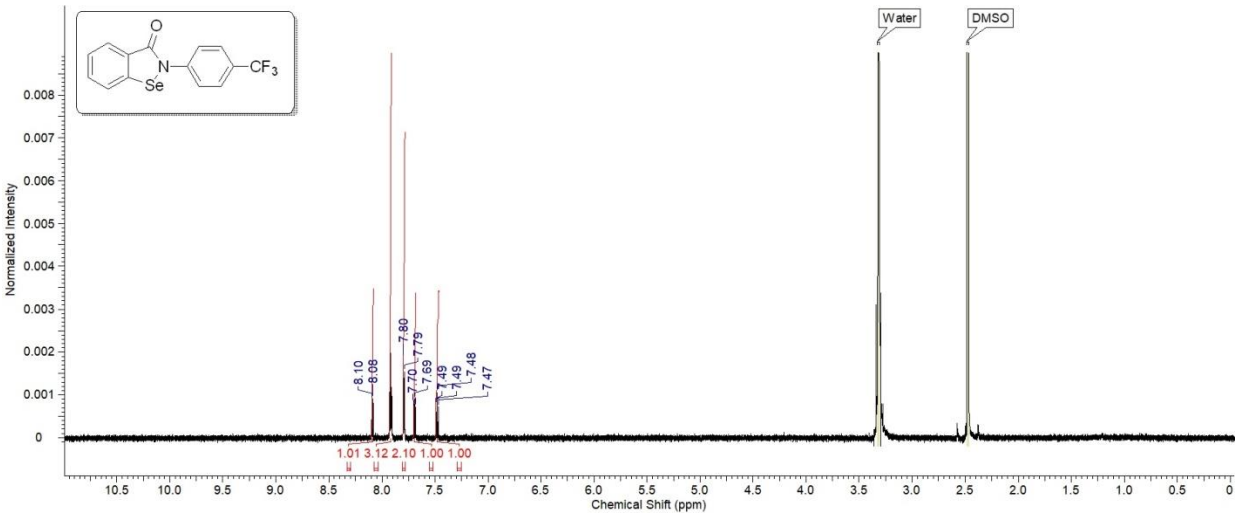
Yield: 46% (Method A); Yield: 90% (Method B); mp 228-230°C;
^{1}H NMR (700 MHz, DMSO) δ = 7.48 (dt, *J*=7.7, 0.7 Hz, 1H$_{ar}$), 7.69 (dt, *J*=8.4, 1.4 Hz, 1H$_{ar}$), 7.78 (d, *J*= 8.4, 2H$_{ar}$), 7.91-7.93 (m, 3H$_{ar}$), 8.09 (d, *J*= 7.7, 1H$_{ar}$) ppm; ^{13}C NMR (176.10 MHz, DMSO) δ = 123.85, 124.74, 125.39, 125.78, 125.96, 126.35, 126.80 (q), 128.61, 128.87, 133.19, 139.16, 144.17, 165.95 (C=O) ppm; ^{77}Se (133.55 MHz, DMSO), δ = 975.57 ppm; IR: 2922, 1624, 1597, 1568, 1513, 1445, 1419, 1320, 1305, 1268, 1192, 1174, 1107, 1070, 1014 cm^{-1}; Elemental Anal. Calcd for $C_{14}H_8F_3NOSe$ (342.97): C, 49.14; H, 2.36, Found: C, 48.95; H, 2.29.

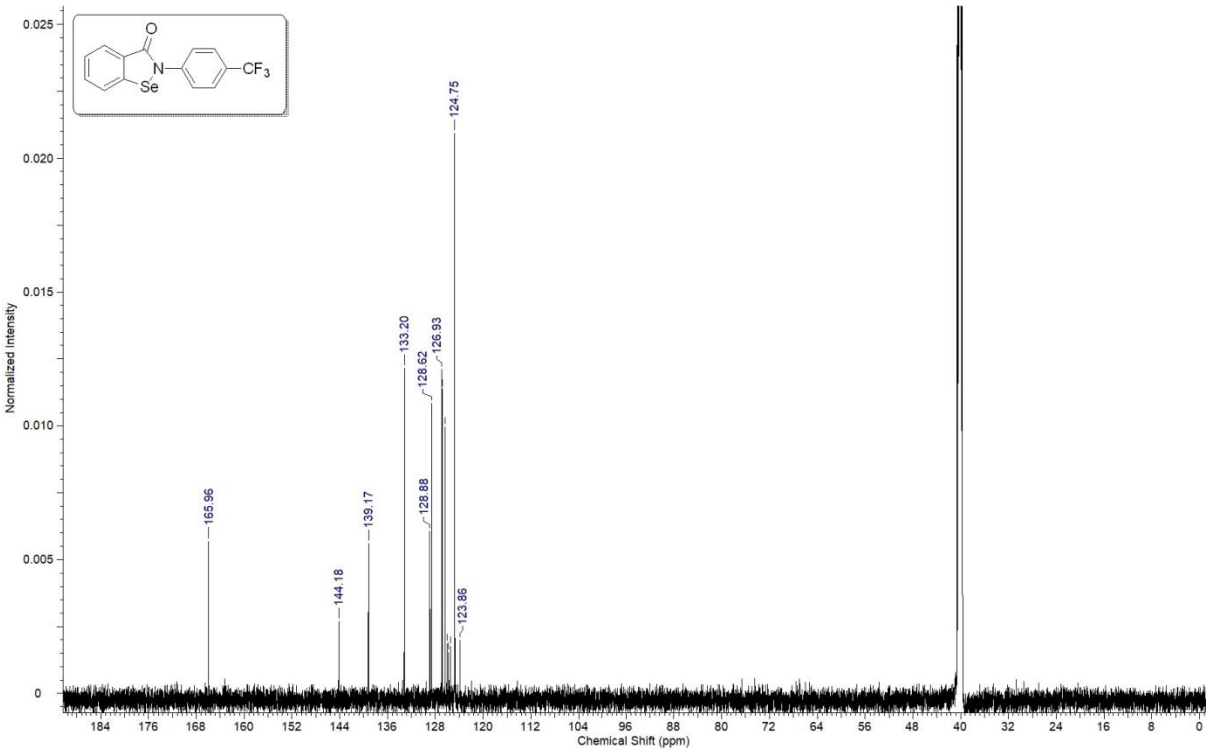
***N*-(2-(Trifluoromethyl)phenyl)-1,2-benzisoselenazol-3(2*H*)-one 22**

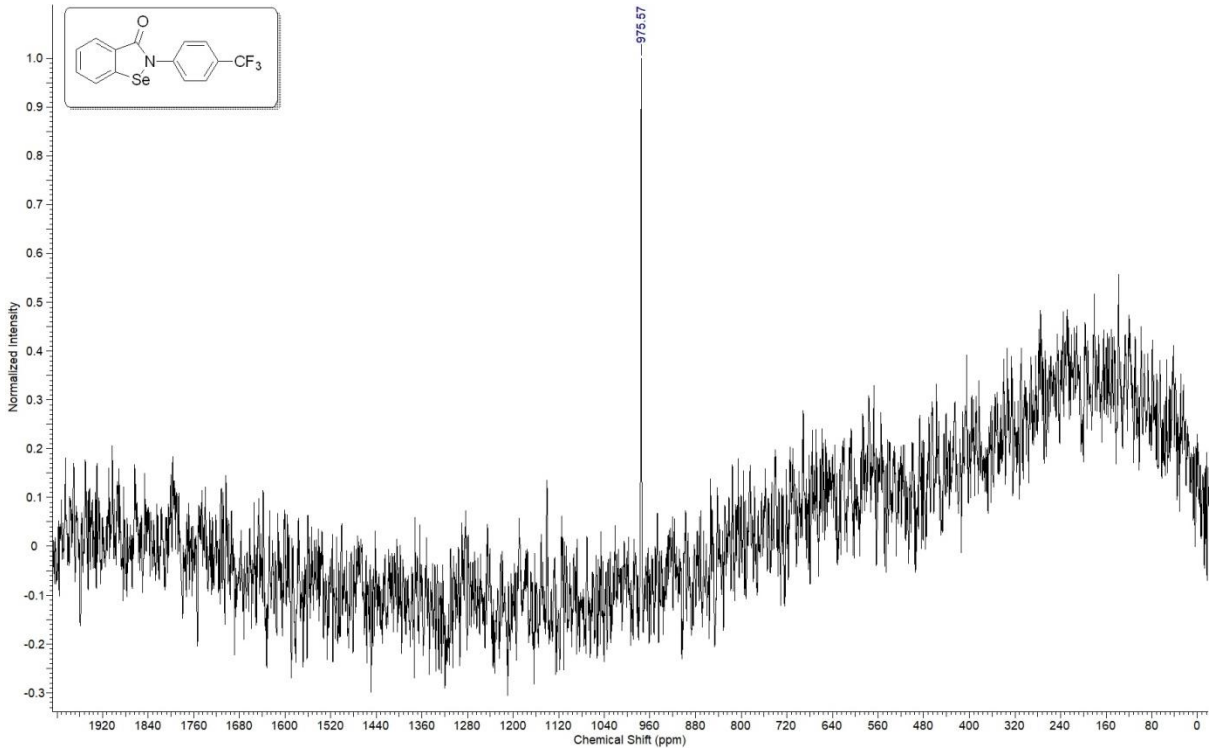
Yield: 41% (Method A); Yield: 89% (Method B); (mp 199-201°C;
^{1}H NMR (700 MHz, DMSO) δ = 7.47 (dt, *J*=7.7, 0.7 Hz, 1H$_{ar}$), 7.49 (d, *J*=7.7, 1H$_{ar}$), 7.65 (t, *J*= 7.7, 1H$_{ar}$), 7.68 (dt, *J*=7.7, 0.7 Hz, 1H$_{ar}$), 7.77 (t, *J*= 7.0, 1H$_{ar}$), 7.85 (d, *J*= 8.4, 2H$_{ar}$), 8.05 (d, *J*= 8.4, 1H$_{ar}$) ppm; ^{13}C NMR (176.10 MHz, DMSO) δ = 123.01, 124.57, 126.31, 126.69, 126.83, 127.64 (q), 128.39, 128.83, 129.00, 129.52, 132.75, 132.96, 134.15, 137.49, 140.90, 167.36 (C=O) ppm; ^{77}Se (133.6 MHz, DMSO), δ = 975.63 ppm; IR: 3064, 1590, 1561, 1496, 1454, 1443, 1347, 1317, 1272, 1260, 1221, 1175, 1164, 1128, 1117, 1109, 1058, 1034, 1025 cm^{1}; Elemental Anal. Calcd for $C_{14}H_8F_3NOSe$ (342.97): C, 49.14; H, 2.36, Found: C, 49.33; H, 2.39.

III. ^{1}H, ^{13}C and ^{77}Se NMR spectra for compounds **21a**, **22a**, **21** and **22**

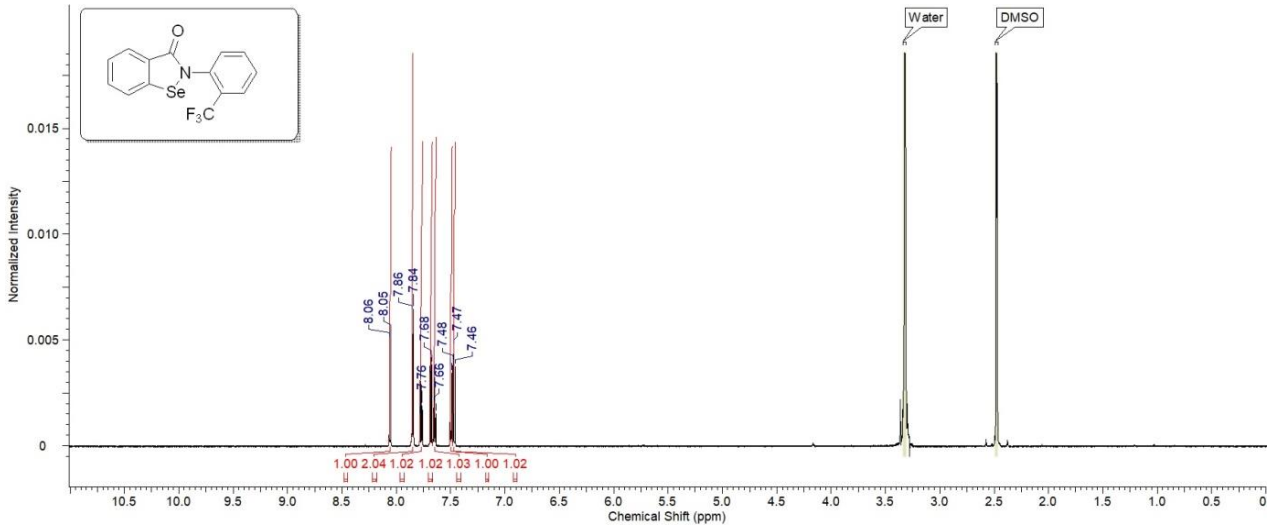
***N*-4-(Trifluoromethyl)phenyl-1,2-benzisoselenazol-3(2*H*)-one 21**

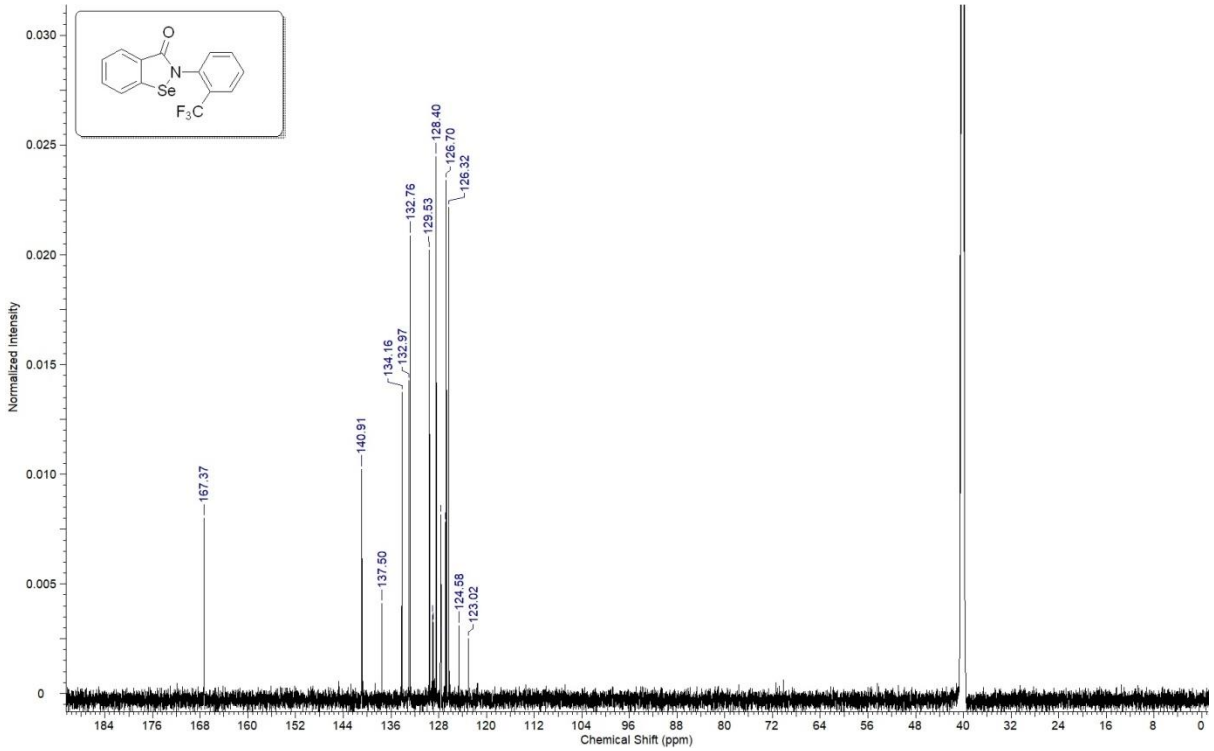


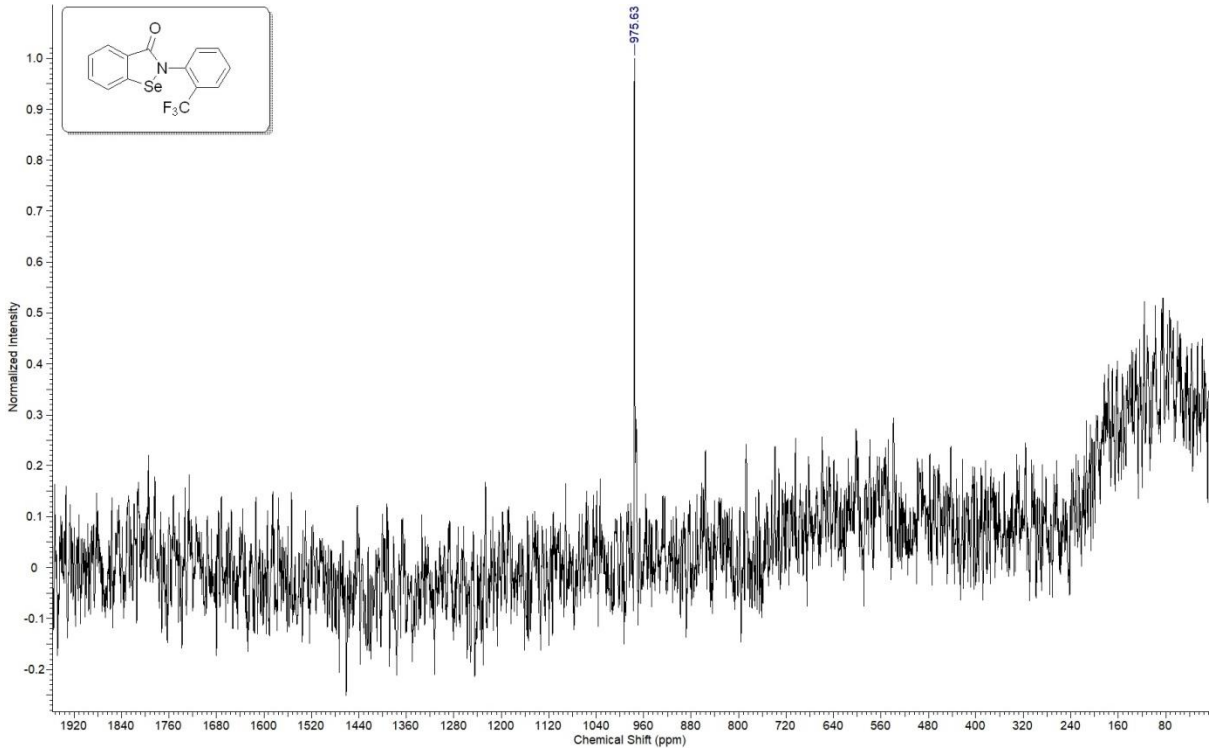




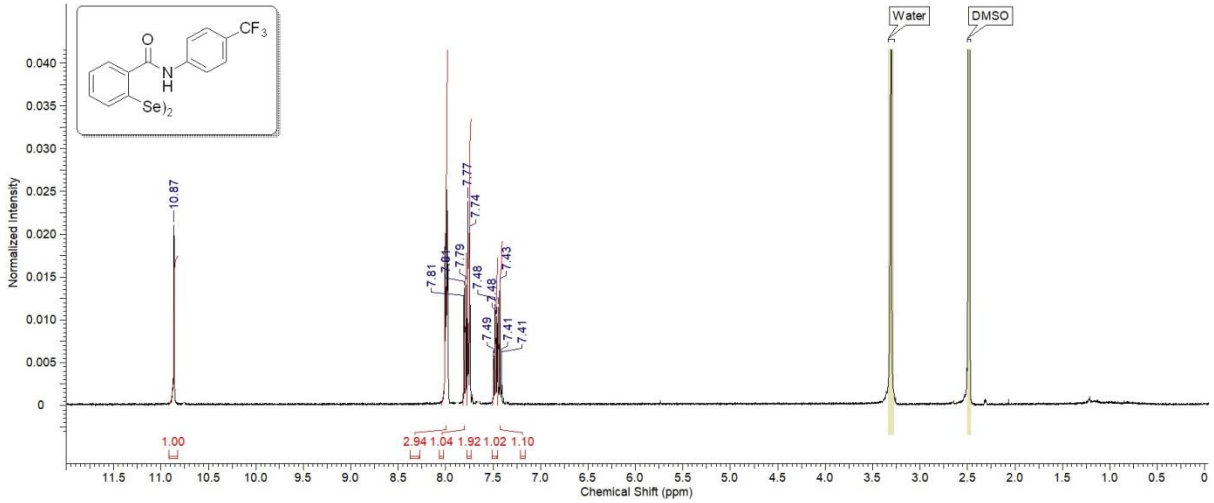
***N*-(2-(Trifluoromethyl)phenyl)-1,2-benzisoselenazol-3(2*H*)-one 22**

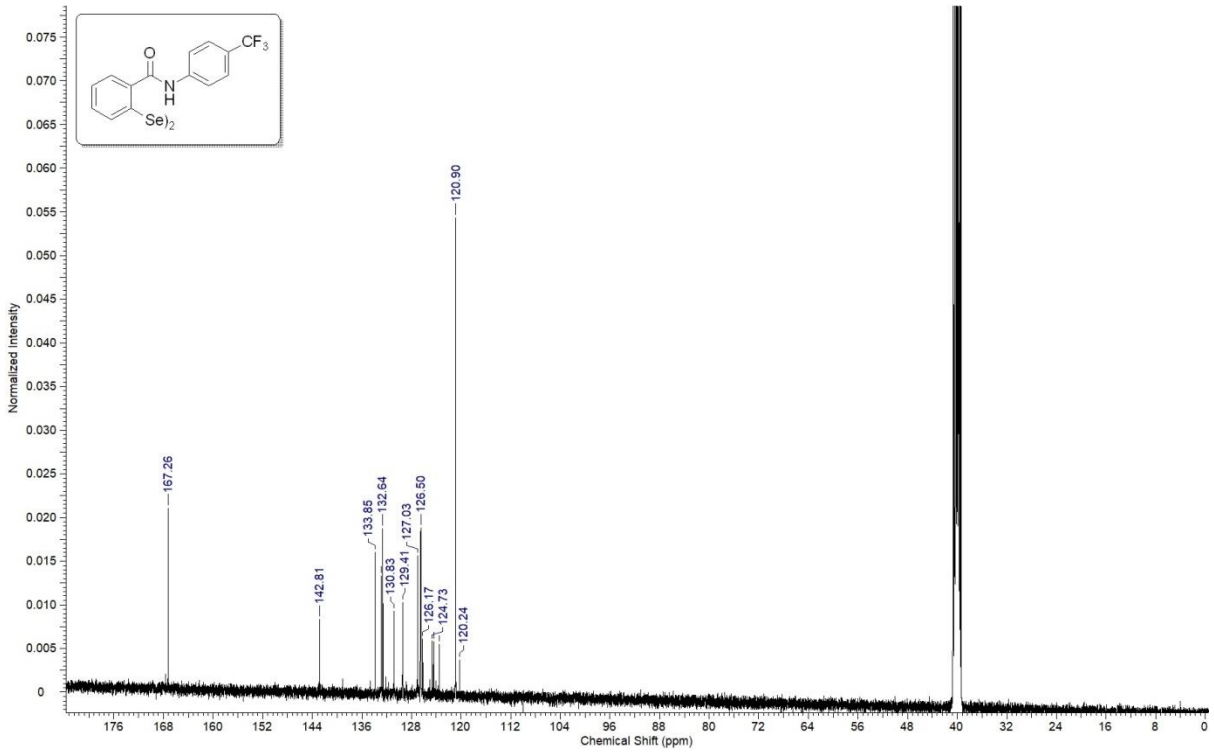


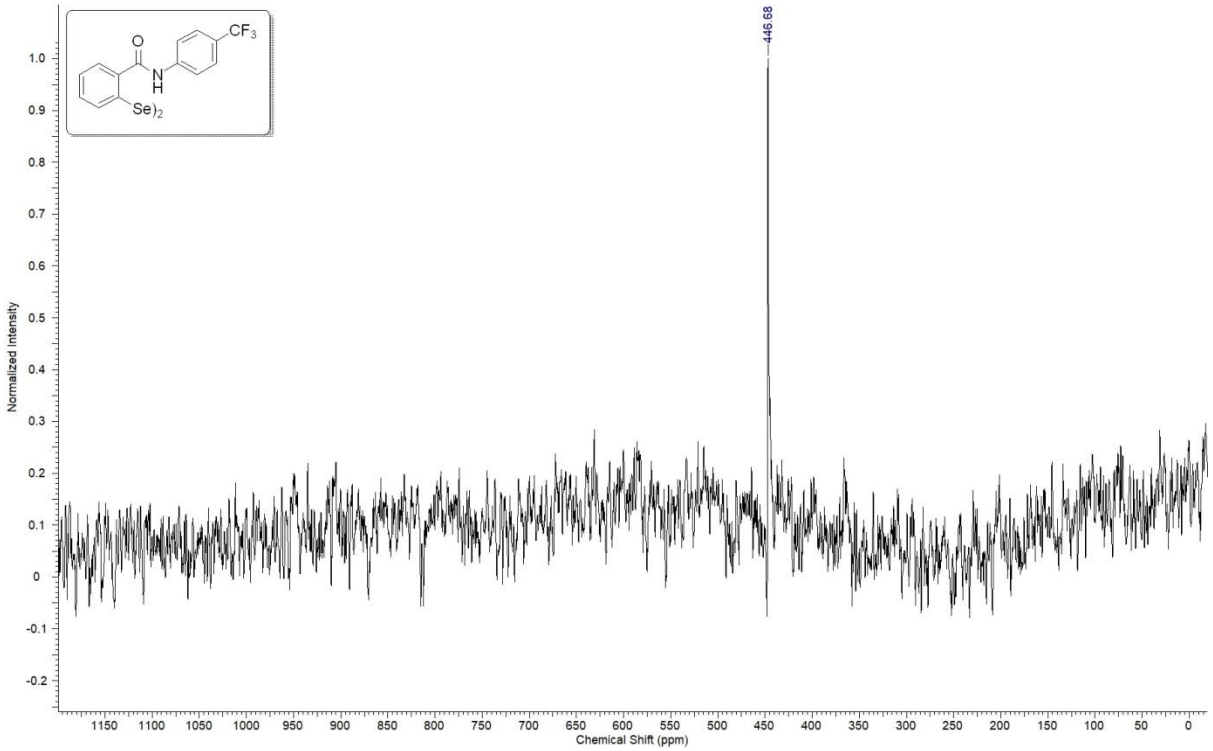

O
N
Se
F3C
167.37
140.91
137.50
134.16
132.97
132.76
129.53
128.40
126.70
126.32
124.58
123.02
Normalized Intensity
Chemical Shift (ppm)


O
N
Se
F3C
975.63
Normalized Intensity
Chemical Shift (ppm)

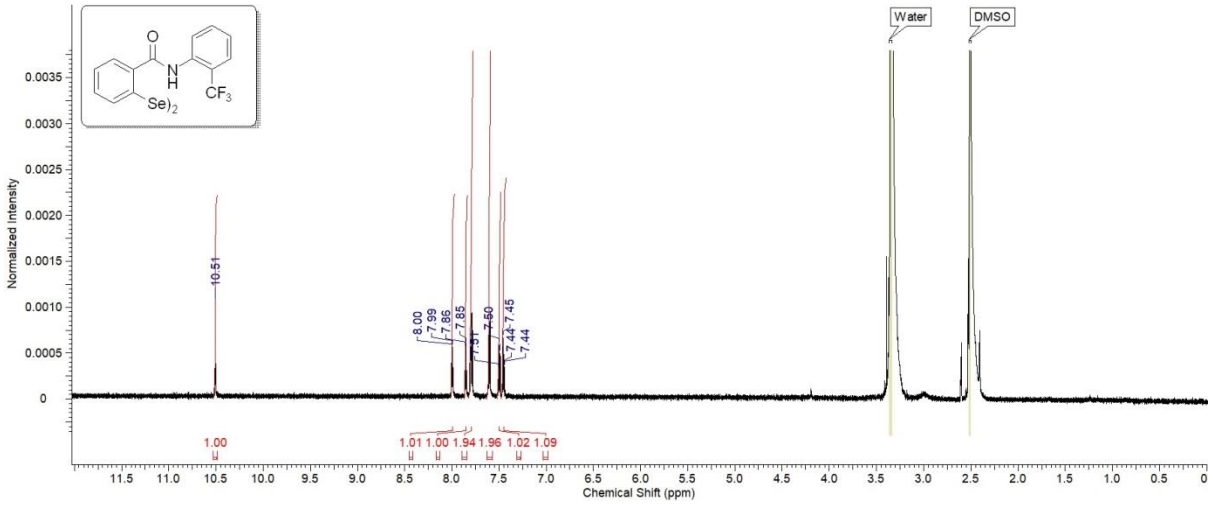
2,2‘-Diselenobis((4-(Trifluoromethyl)phenyl)benzamide) 21a

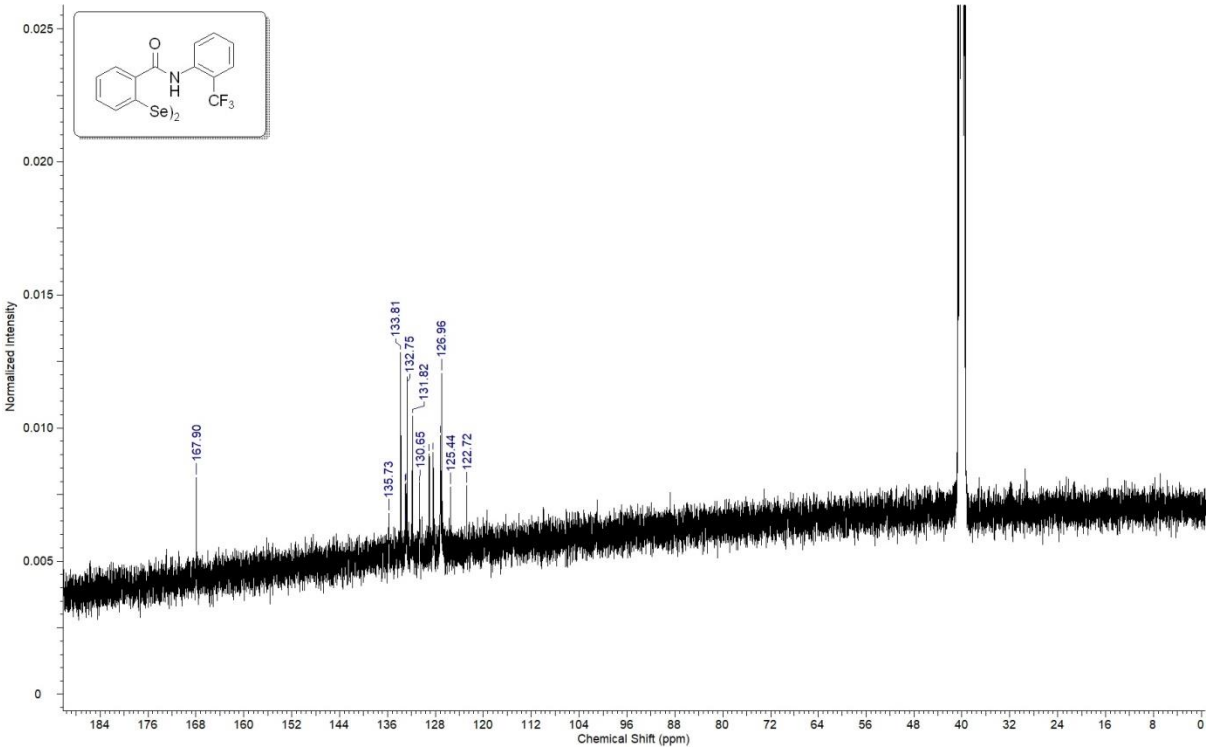


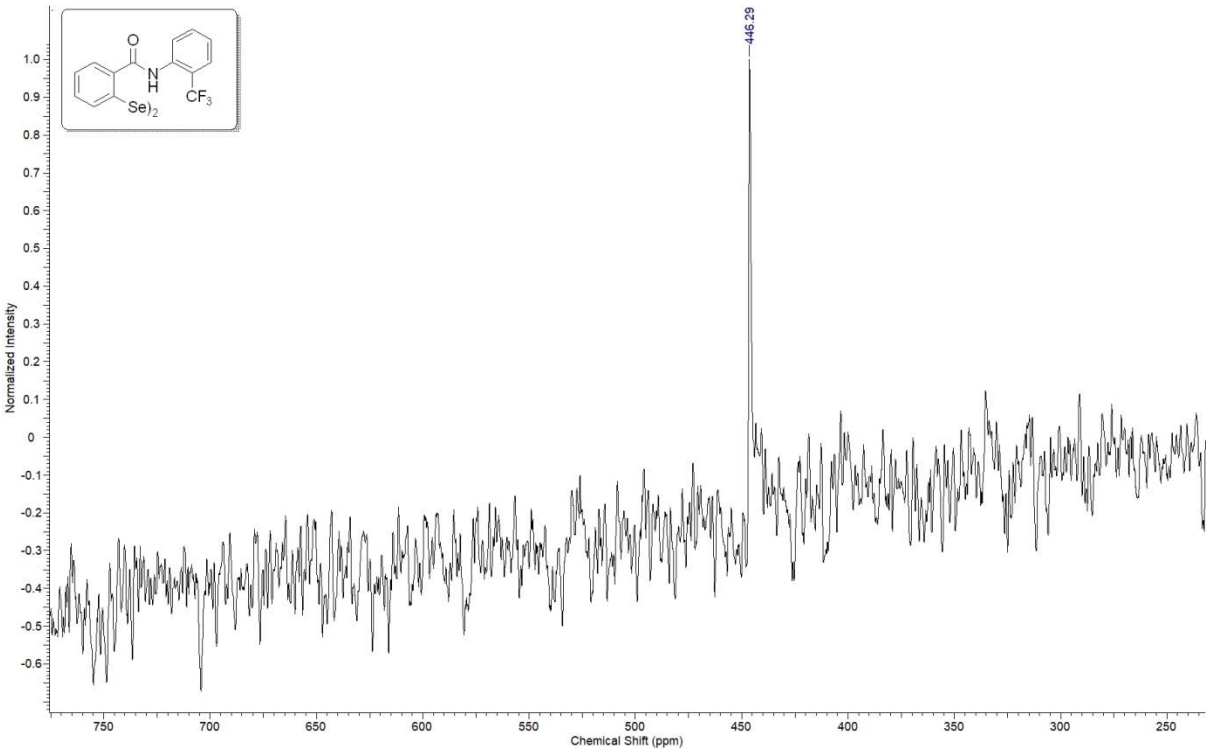




2,2‘-Diselenobis((2-(Trifluoromethyl)phenyl)benzamide) 22a

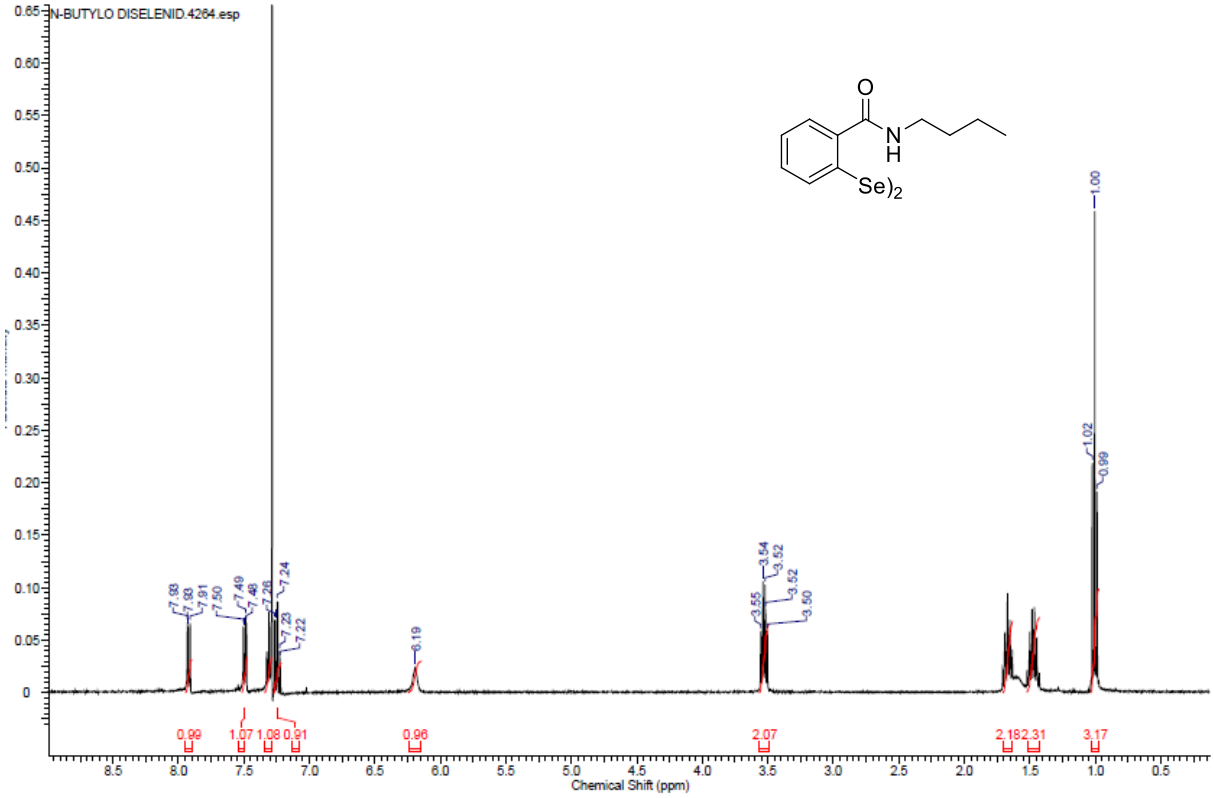



Normalized Intensity
Chemical Shift (ppm)
167.90
135.73
133.81
132.75
131.82
130.65
126.96
125.44
122.72

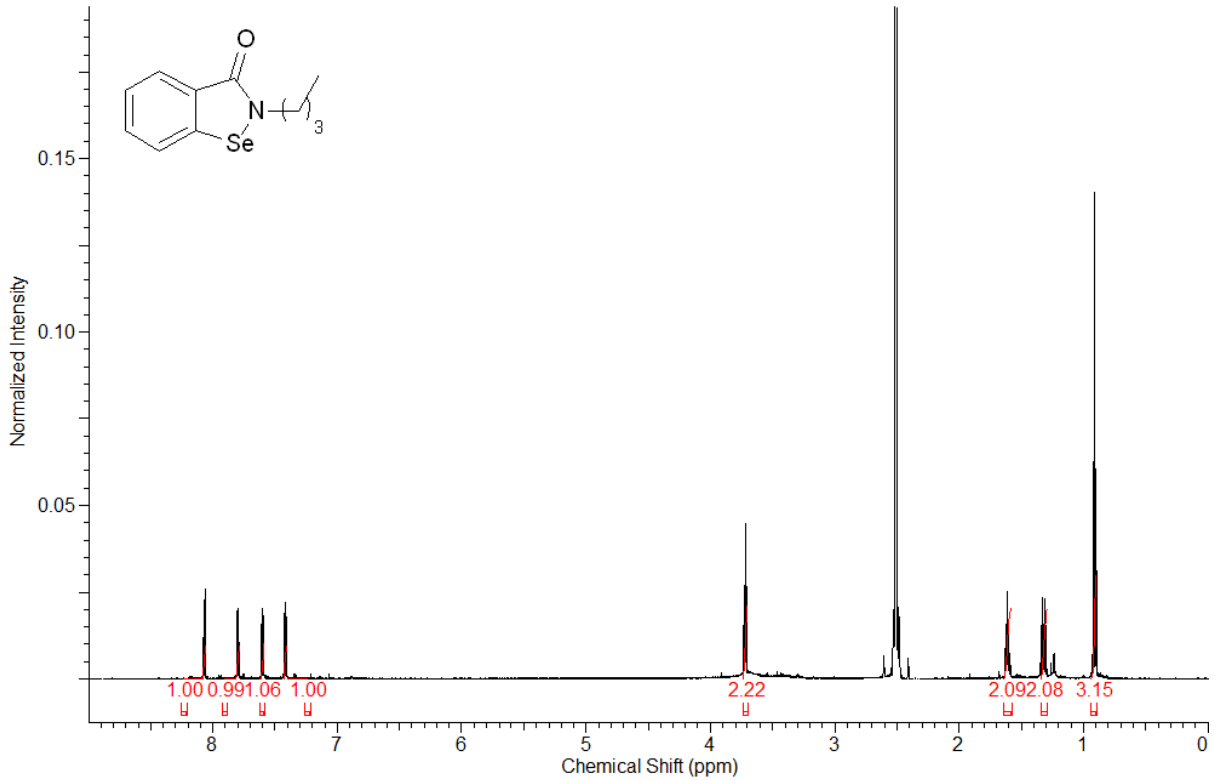

Normalized Intensity
Chemical Shift (ppm)
446.29

IV. ^{1}H NMR spectra for compounds **10**-**20** and **12a**-**20a**.

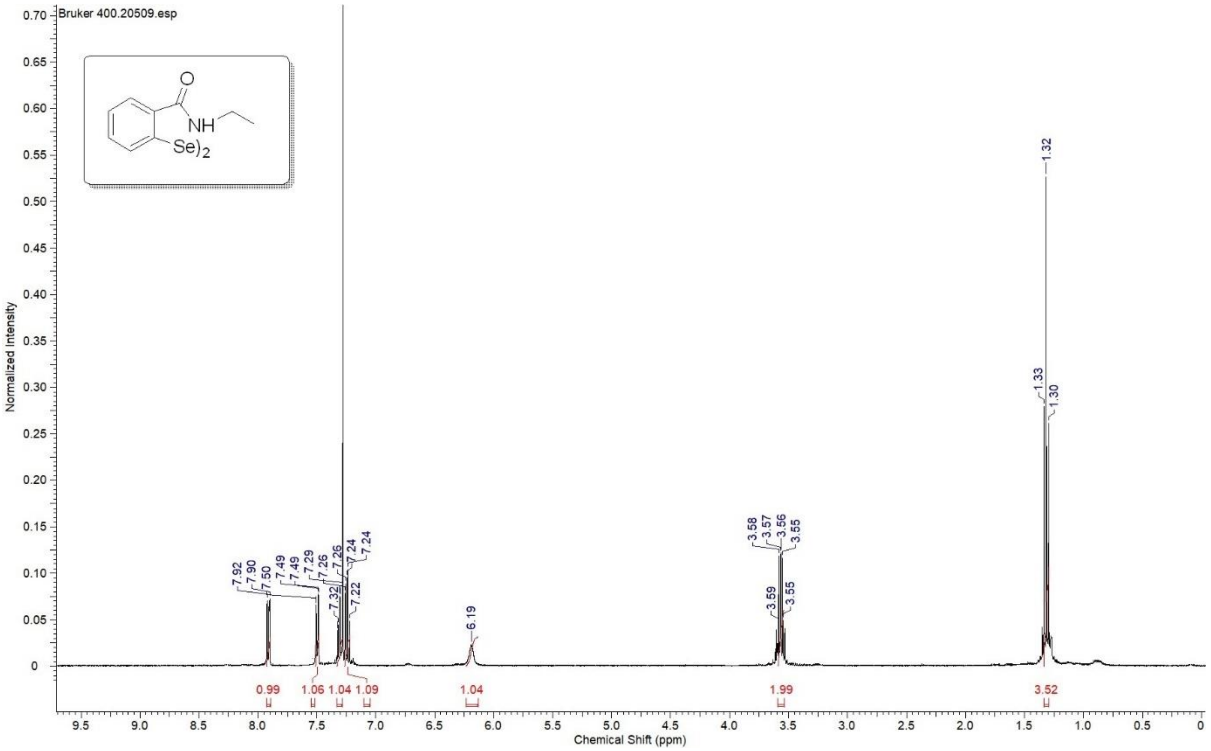
2,2'-Diselenobis(*N*-butylbenzamide) 10[1]



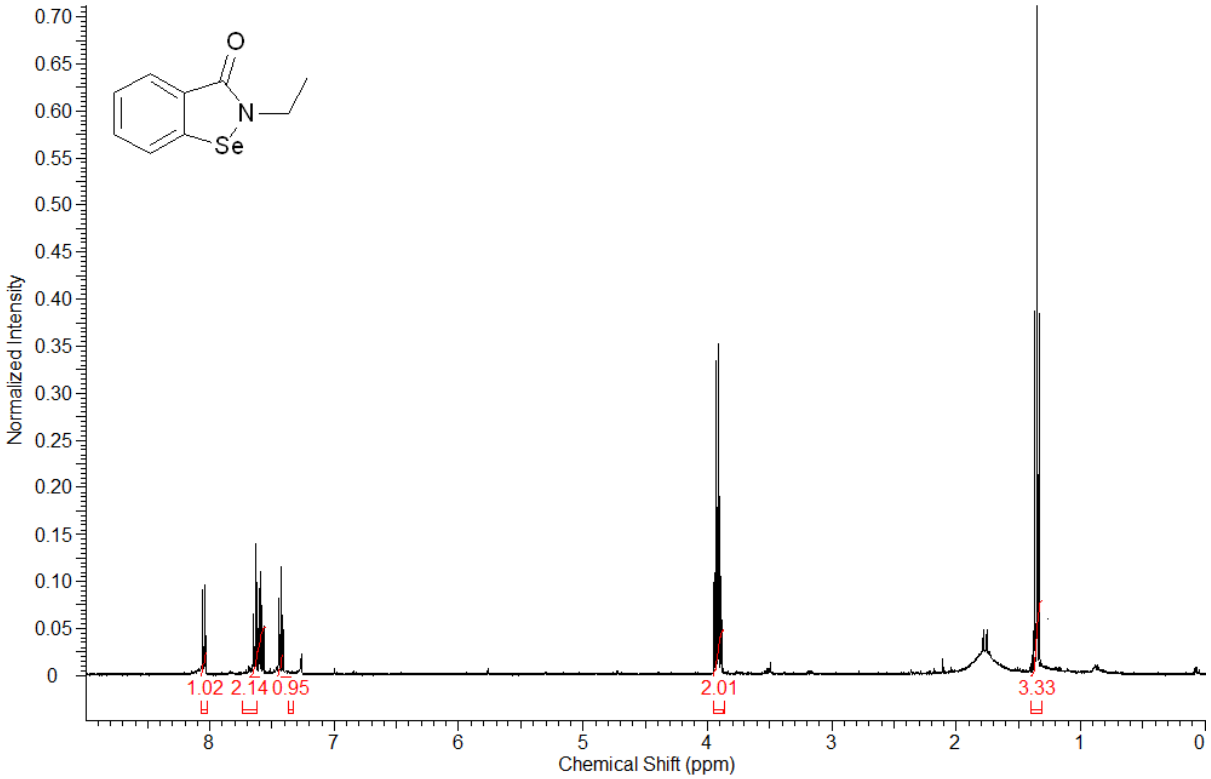
***N*-buthyl-1,2-benzisoselenazol-3(2*H*)-one 11[2]**



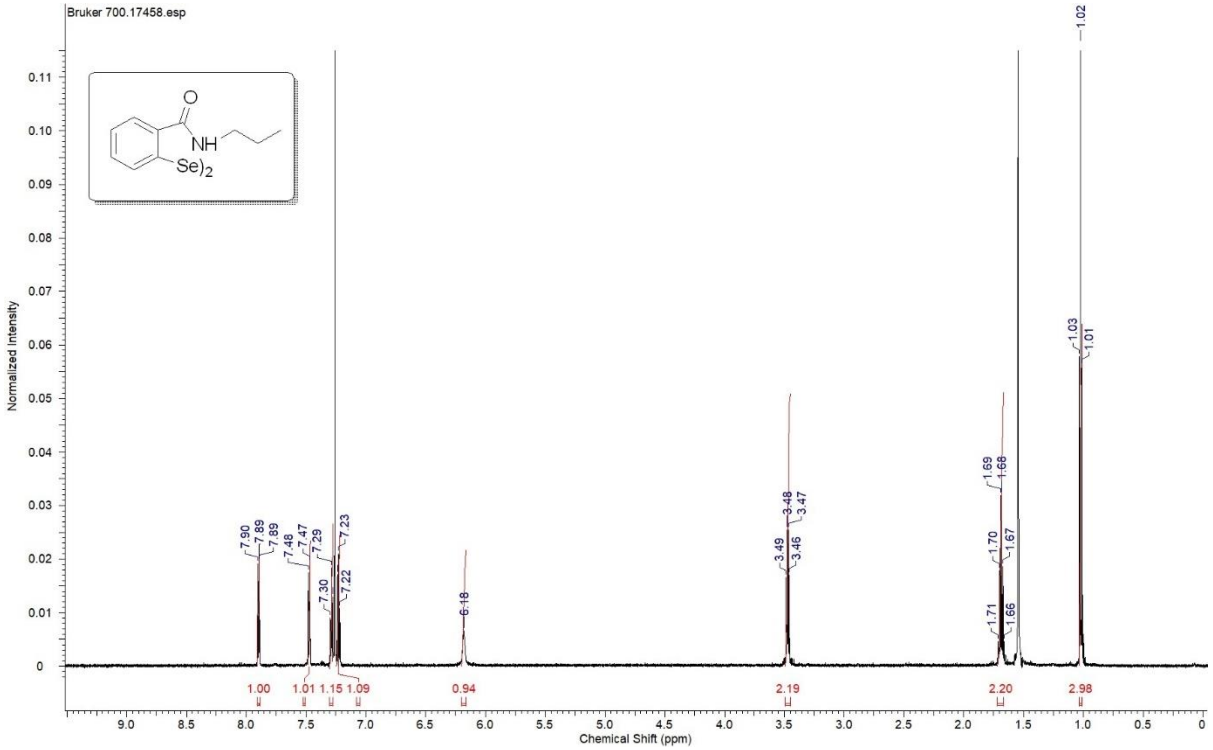
2,2'-Diselenobis(*N*-ethylbenzamide) 12a[3]



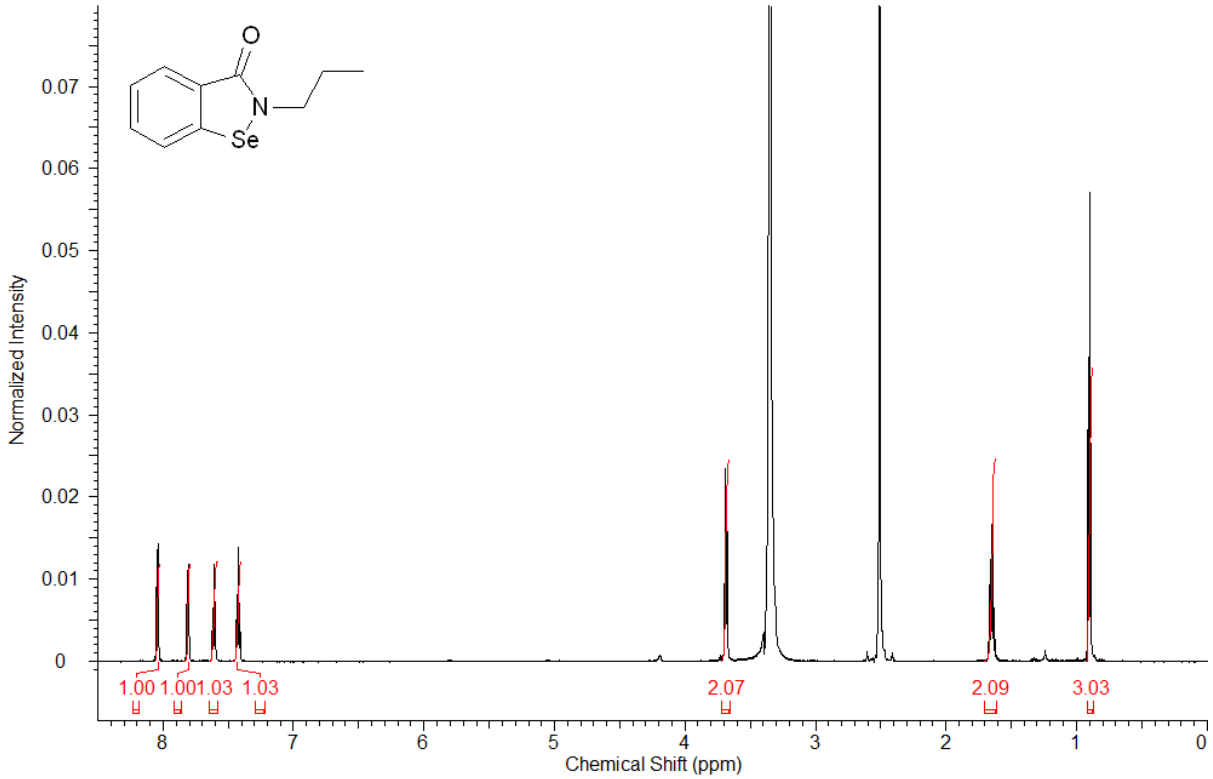
***N*-ethyl-1,2-benzisoselenazol-3(2*H*)-one 12[4]**



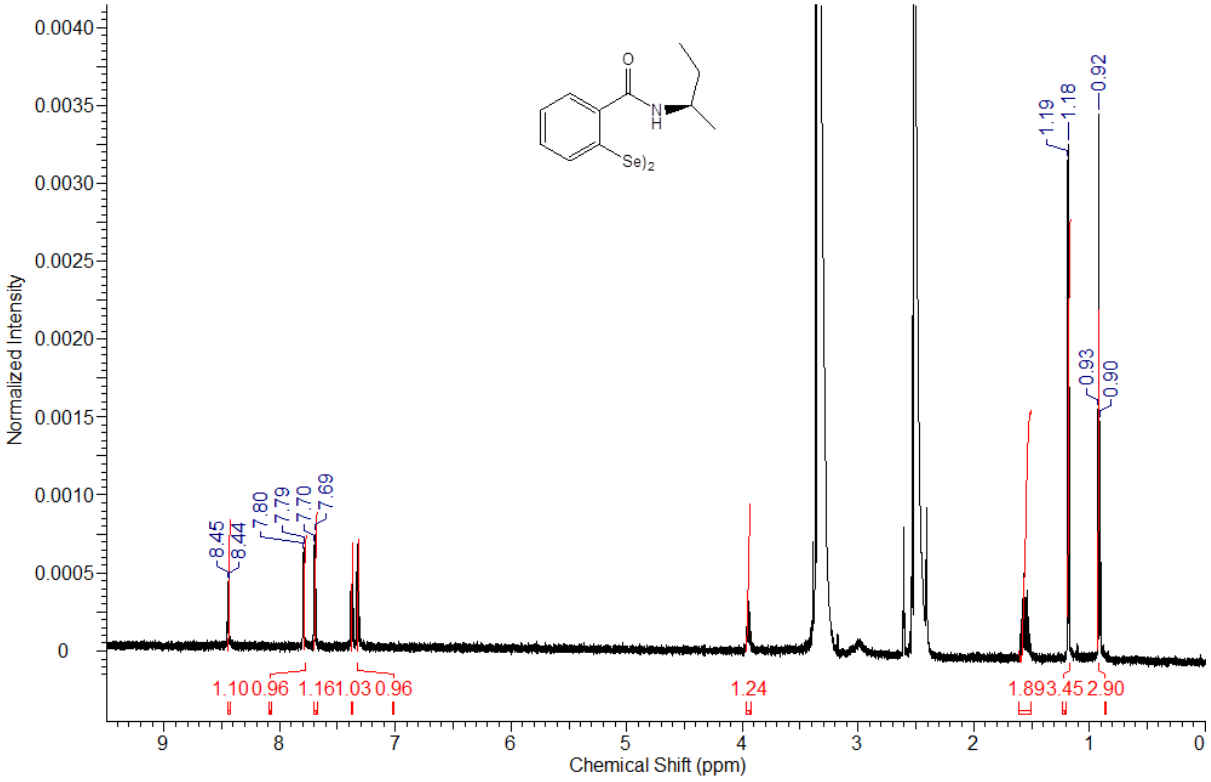
2,2'-Diselenobis(*N*-propylbenzamide) 13a[3]



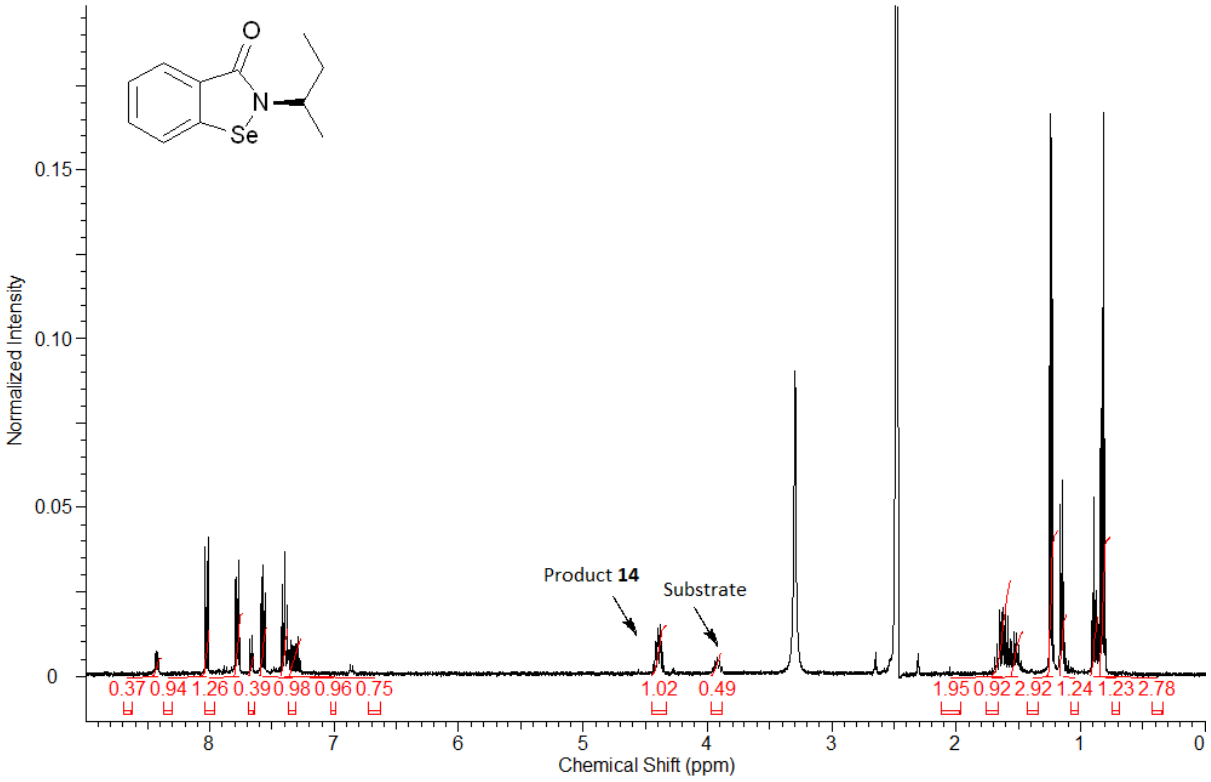
***N*-propyl-1,2-benzisoselenazol-3(2*H*)-one 13[4]**



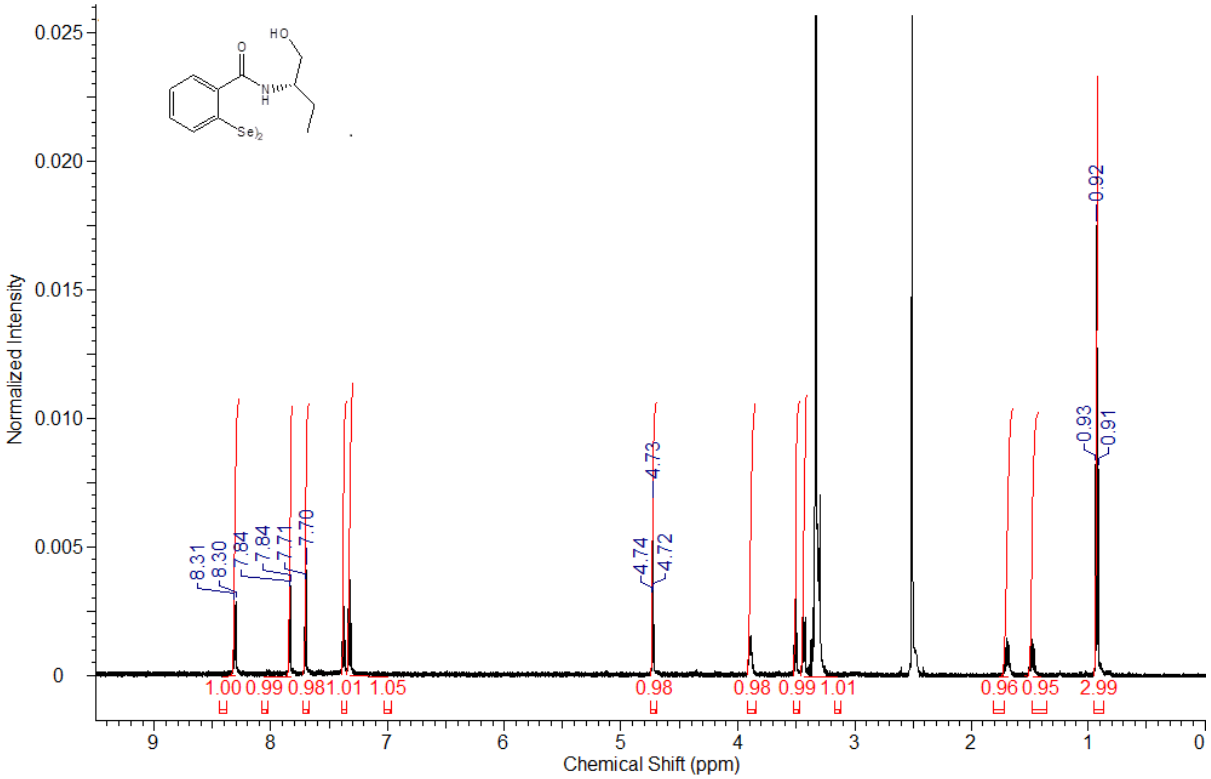
2,2'-diselenobis[*N*-(*R*)-(-)-*sec*-butylbezamide 14a[5]



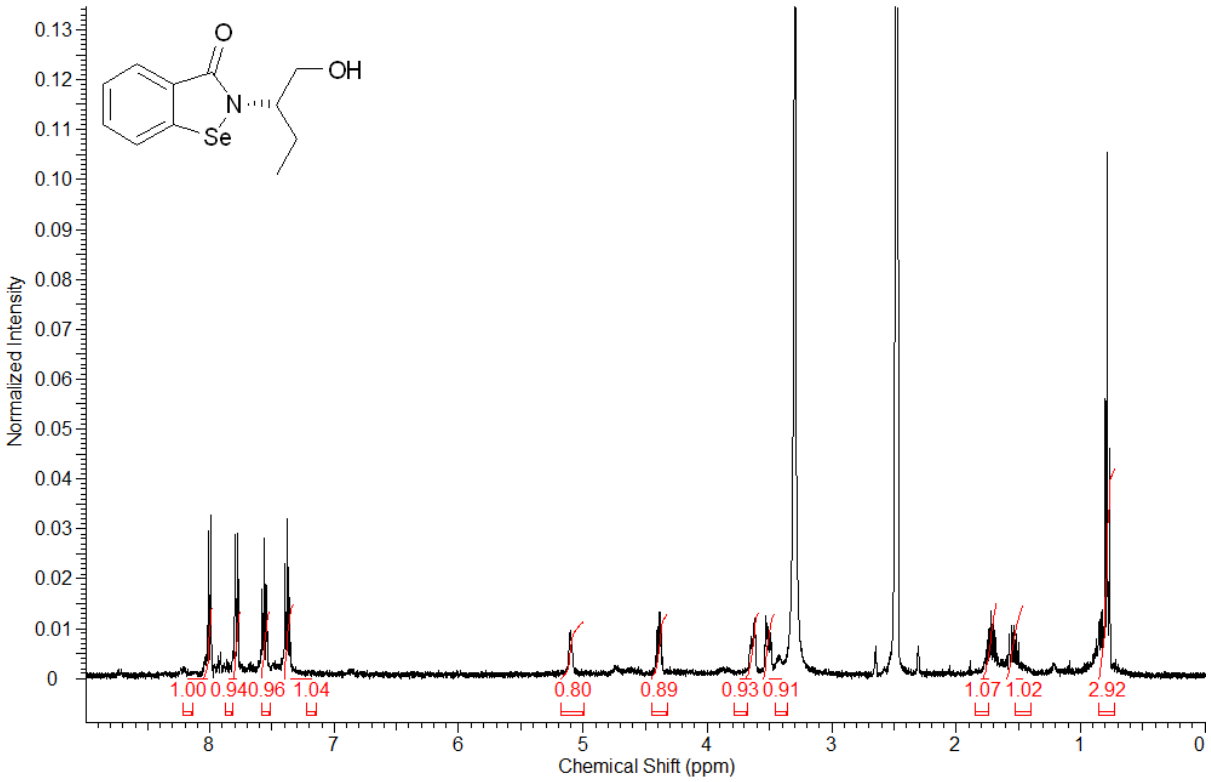
***N*-[(*R*)-(-)-*sec*-butyl]-1,2-benzisoselenazol-3(2*H*)-one 14[5]**



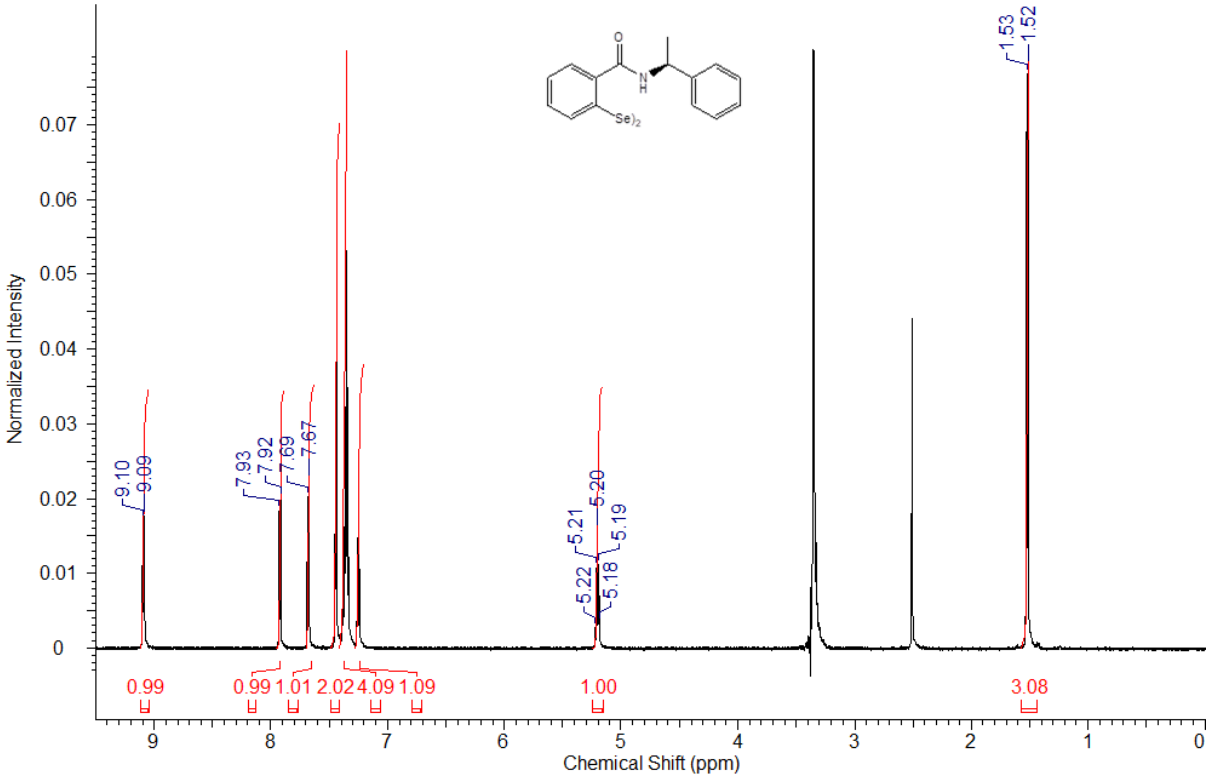
2,2'-diselenobis[*N*-(*S*)-(+)-1-hydroxy-2-butanylbezamide] 15a[5]



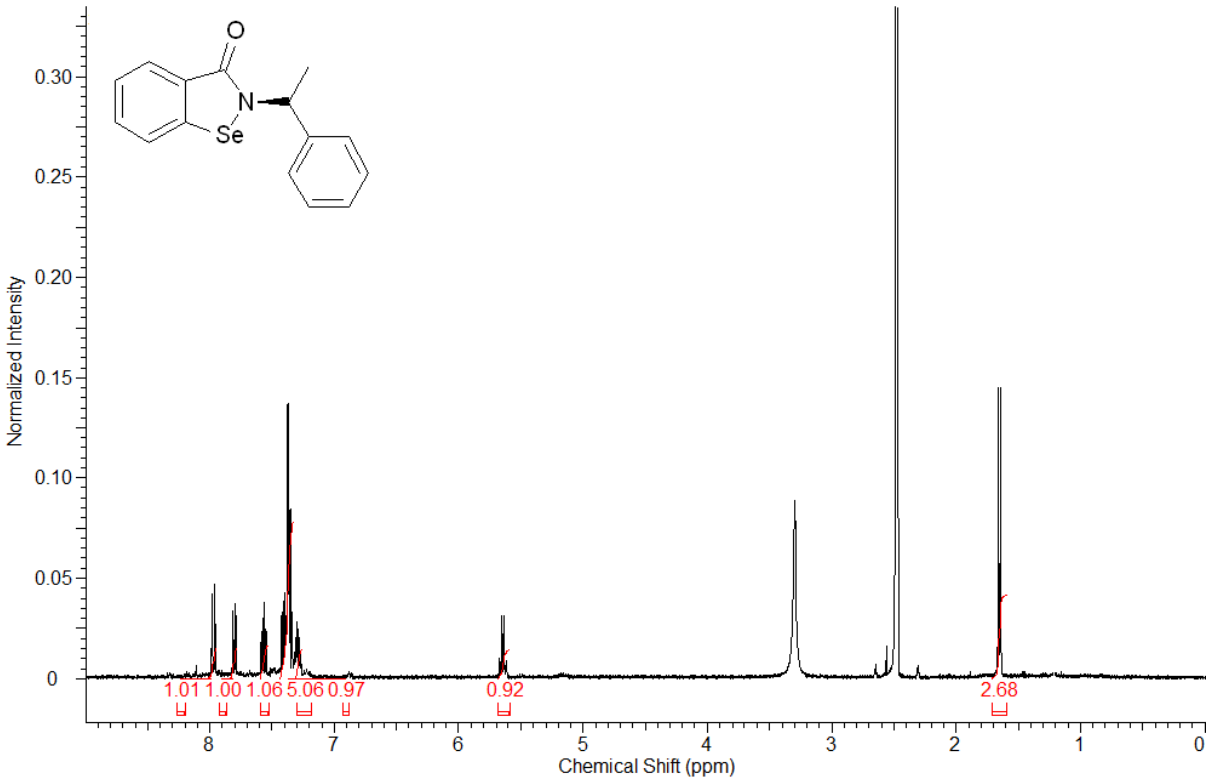
***N*-[(*S*)-(+)-1-hydroxy-2-butanyl]-1,2-benzisoselenazol-3(2*H*)-one 15[5]**



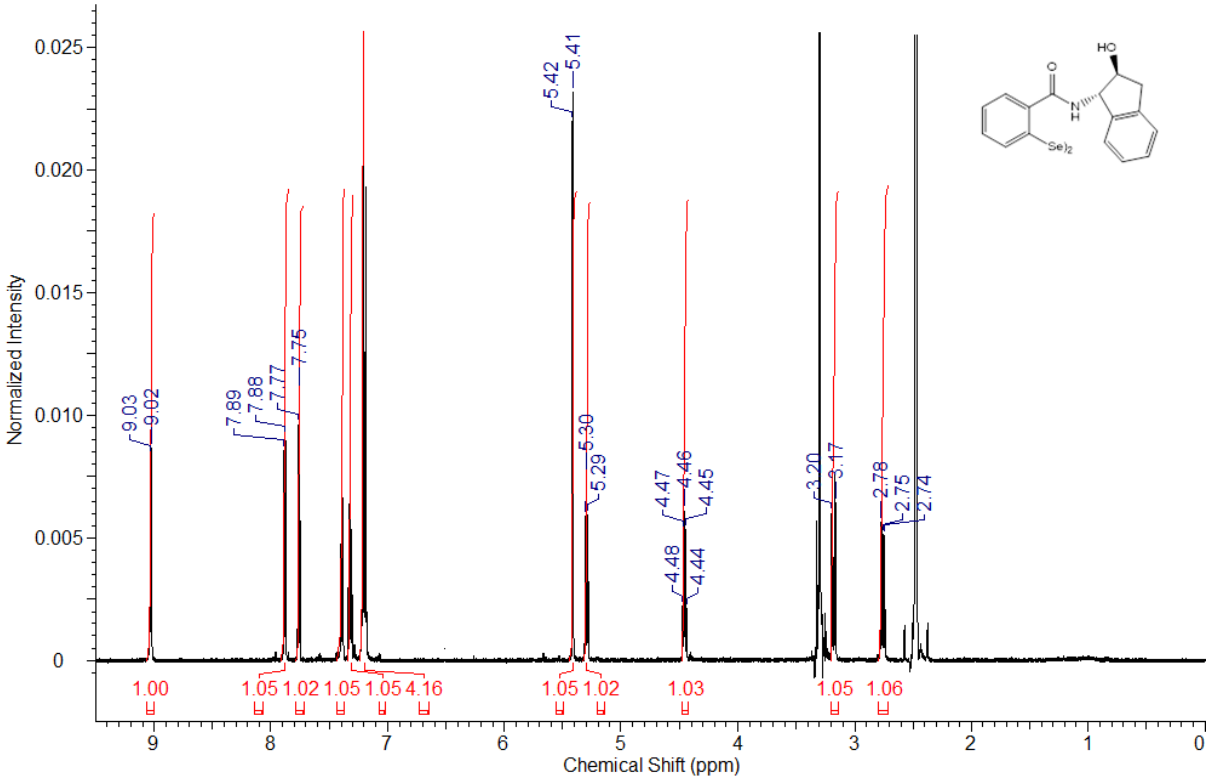
2,2'-diselenobis[*N*-(*S*)-(-)-α-methylbenzylbezamide]16a[5]



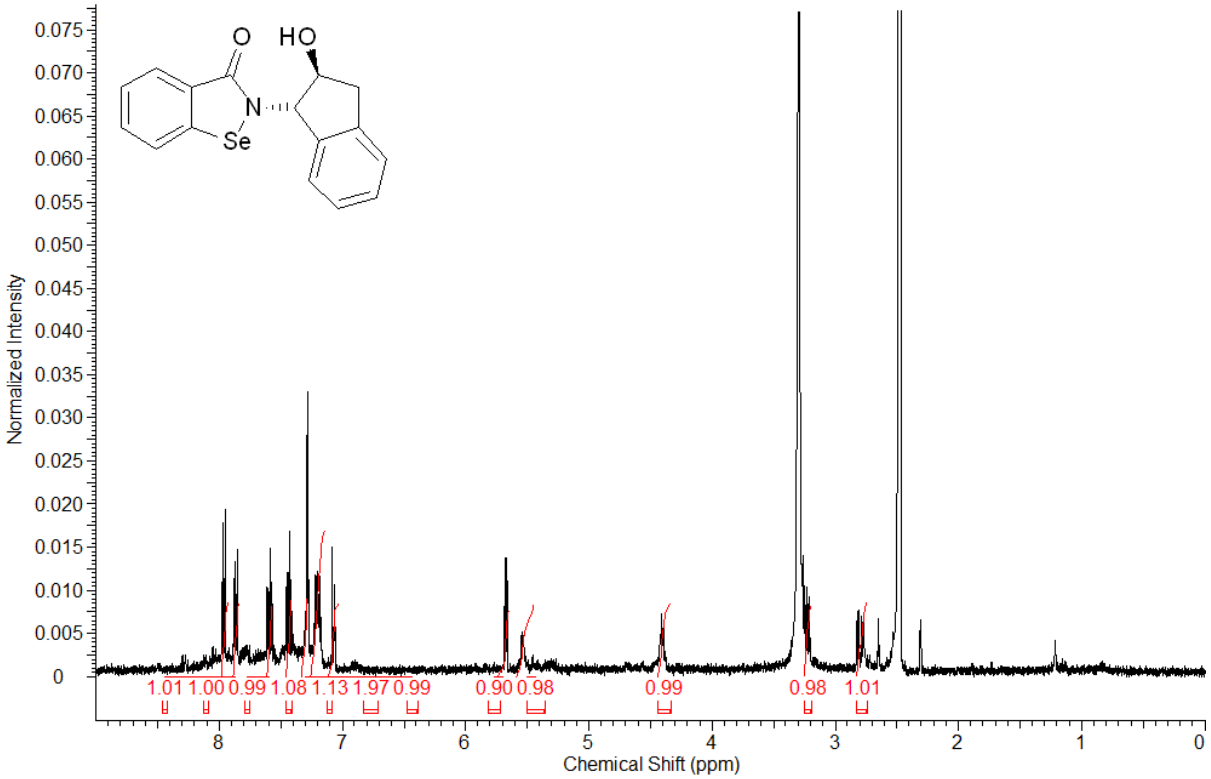
***N*-[(*S*)-(-)-α-methylbenzyl]-1,2-benzisoselenazol-3(2*H*)-one 16[5]**



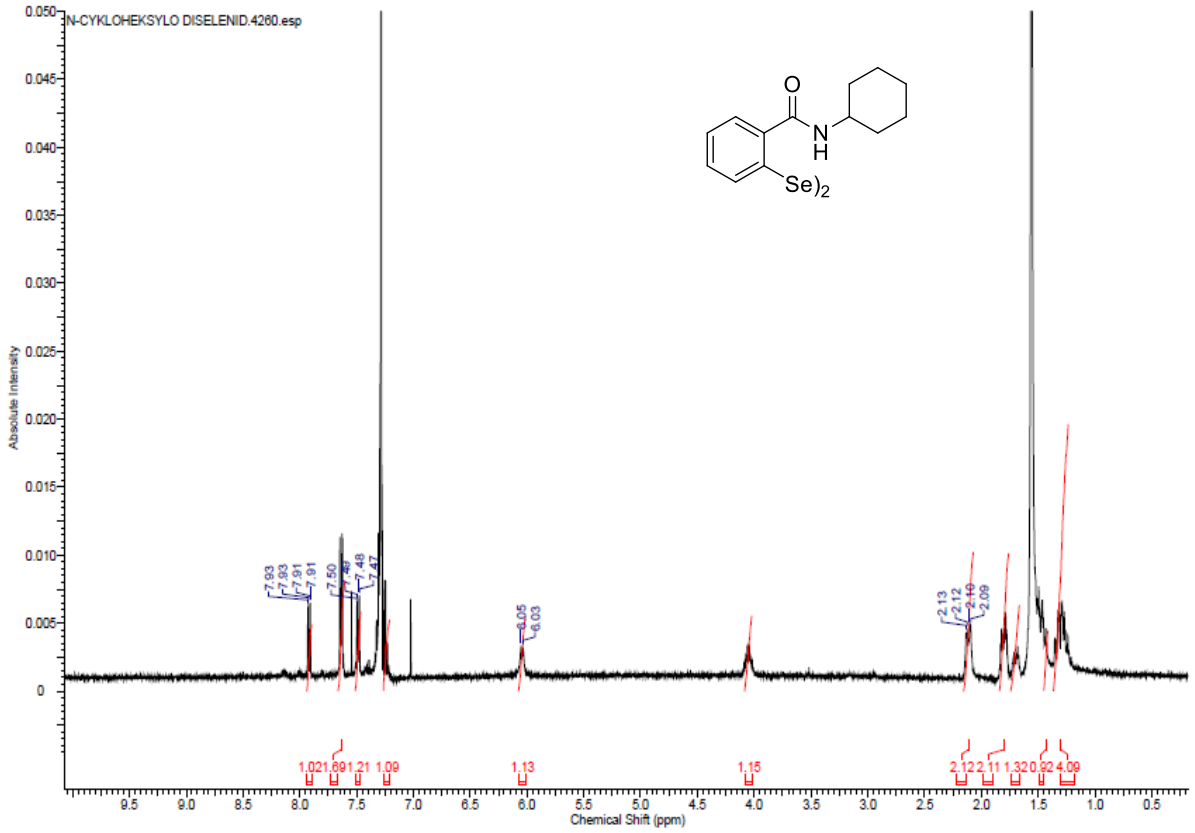
2,2'-diselenobis[*N*-(*1S,2S*)-(-)-*trans*-2-hydroxy-1-indanylbezamide] 17a[5]



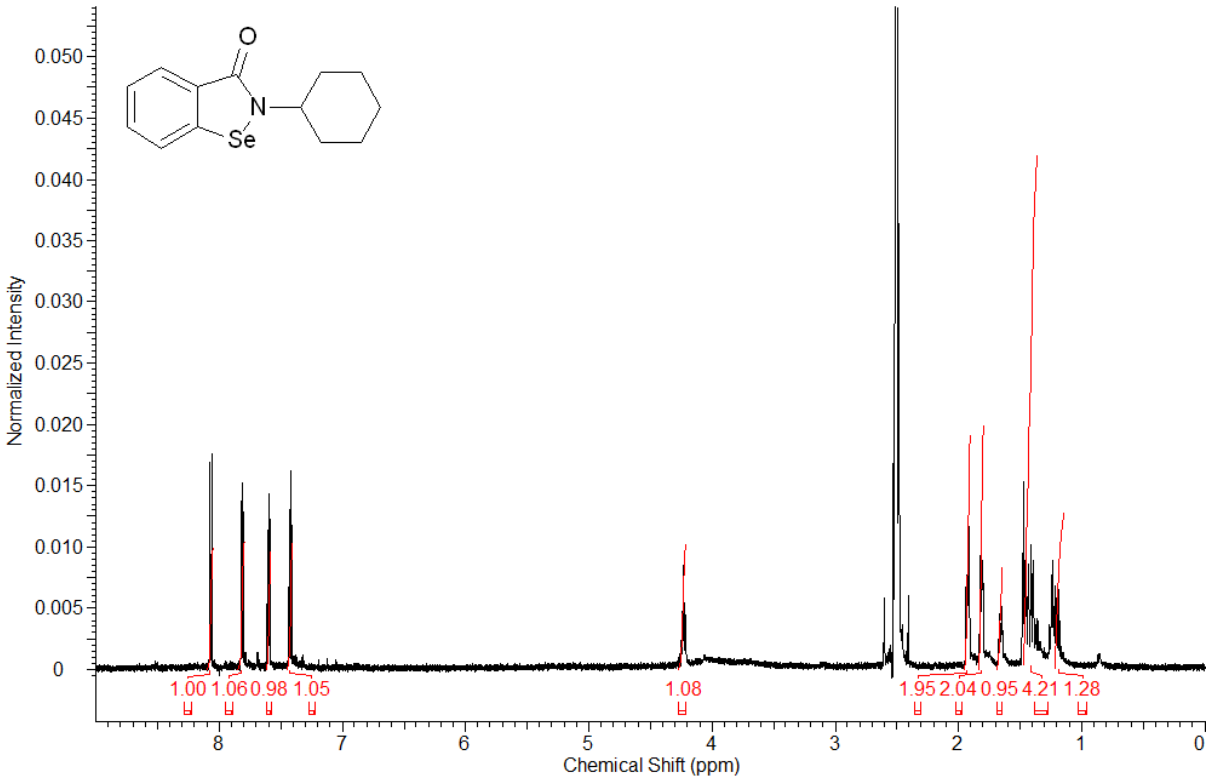
***N*-[(*1S,2S*)-(+)-*trans*-2-hydroxy-1-indanyl]-1,2-benzisoselenazol-3(2*H*)-one 17[5]**



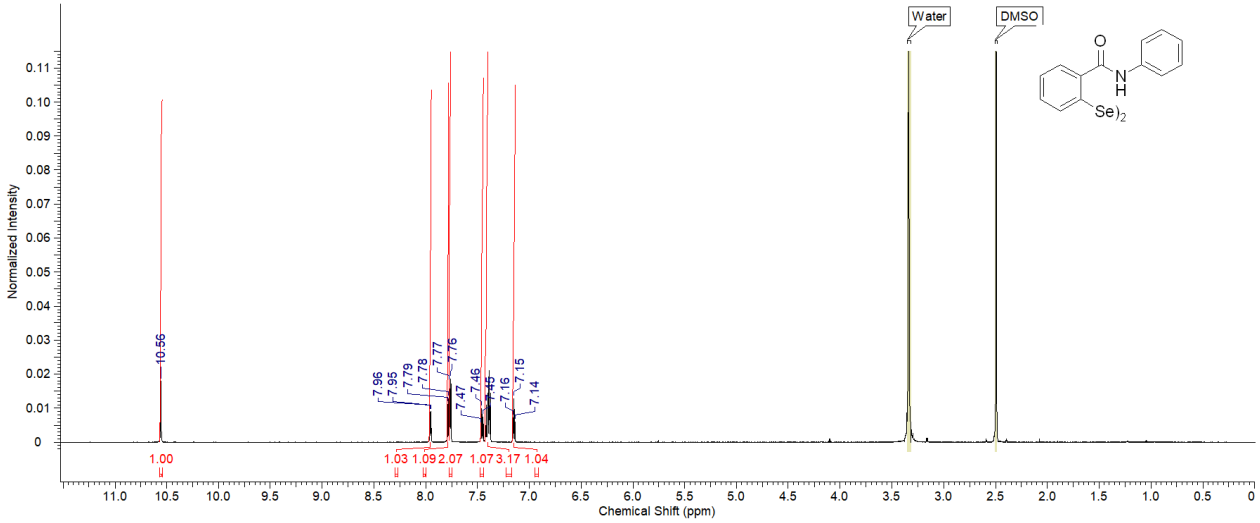
2,2'-Diselenobis(*N*-cyclohexylbenzamide) 18a[1]



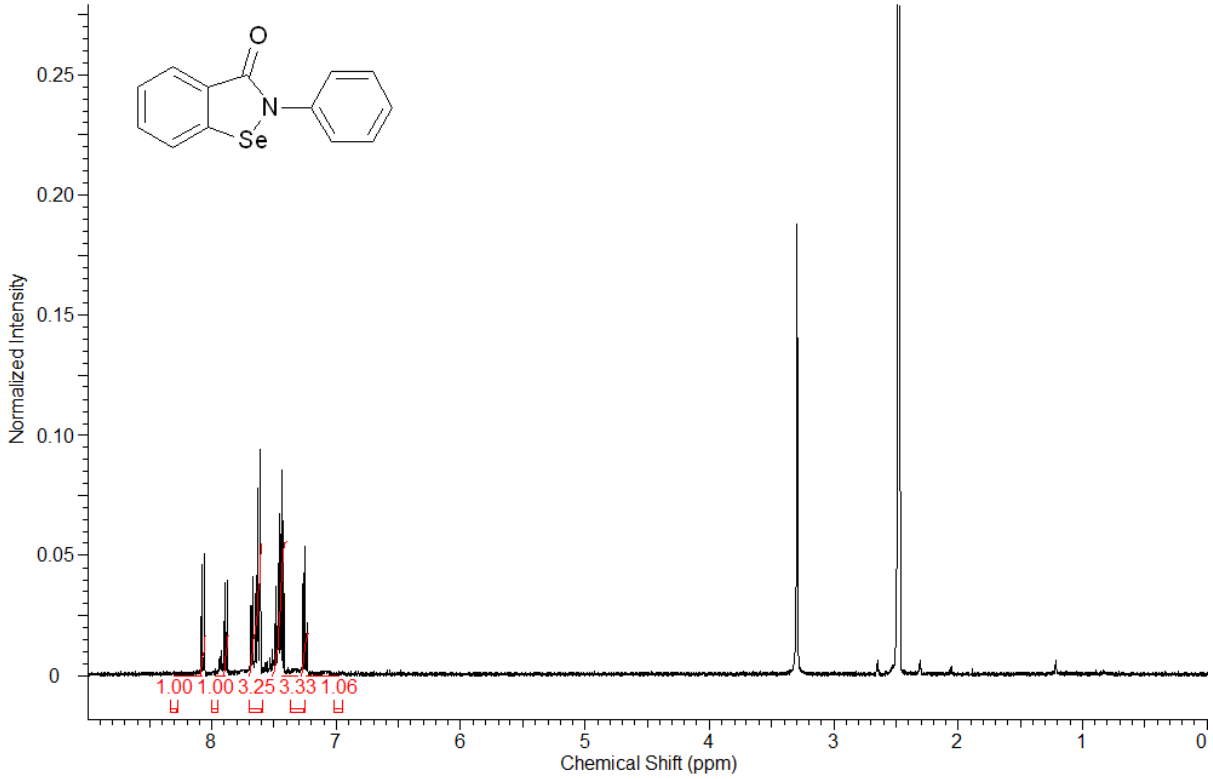
***N*-cyclohexyl-1,2-benzisoselenazol-3(2*H*)-one 18[2]**



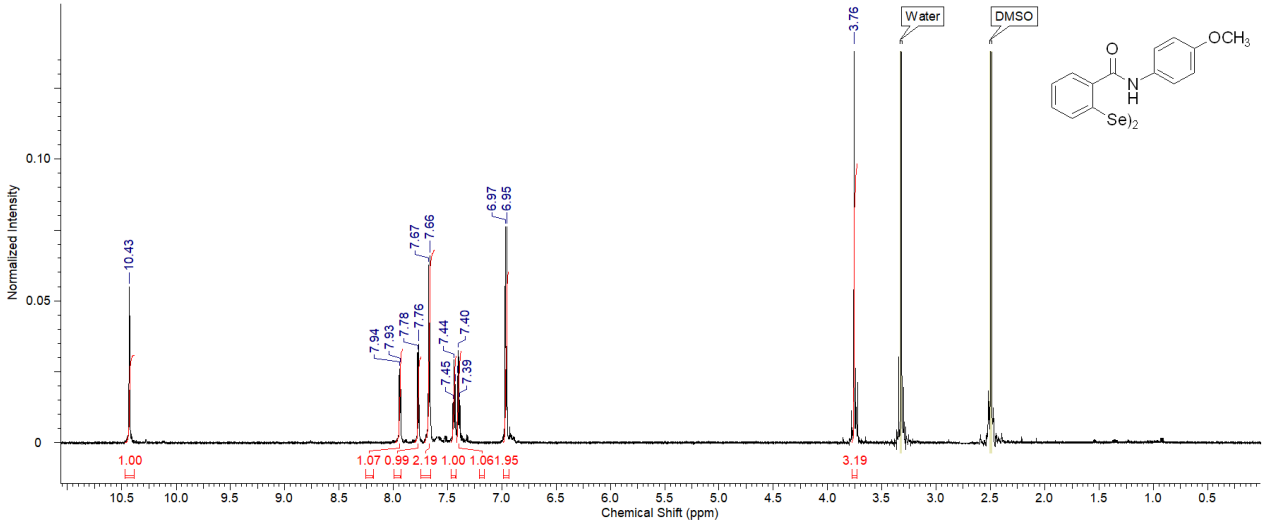
2,2'-Diselenobis(*N*-phenylbenzamide) 19a[6]



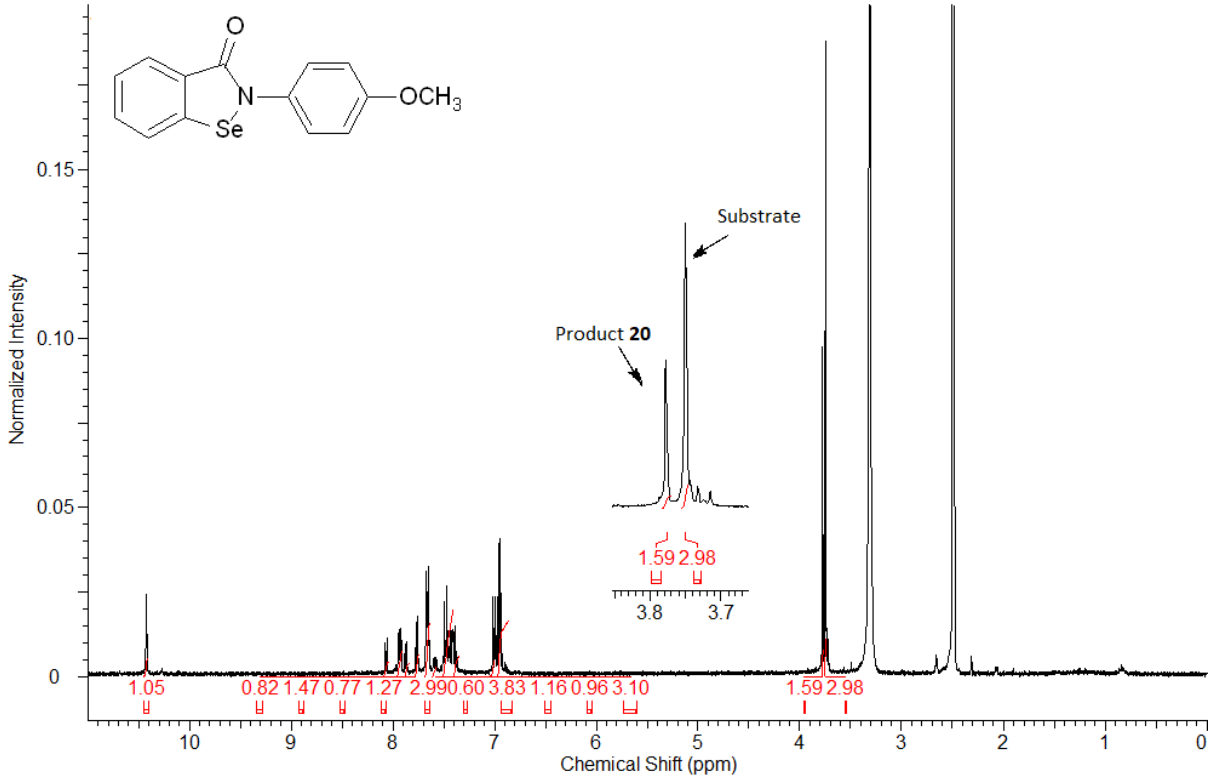
***N*-phenyl-1,2-benzisoselenazol-3(2*H*)-one 19[6]**



2,2'-Diselenobis(*N*-(4-methoxyphenyl)benzamide) 20a[6]



***N*-(4-methoxyphenyl)-1,2-benzisoselenazol-3(2*H*)-one 20[6]**



V. References

1. A. J. Pacuła, K. B. Kaczor, A. Wojtowicz, J. Antosiewicz, A. Janecka, A. Długosz, T. Janecki, J. Scianowski, *Bioorg. Med. Chem*., **2017**, *25*, 126–131.

2. A. J. Pacuła, J. Ścianowski and K. B. Aleksandrzak, *RSC Adv.* **2014**, *4*, 48959–48962.

3. M. Pietka-Ottlik, P. Potaczek, E. Piasecki, J. Mlochowski, *Molecules* **2010**, *15*, 8214-8228.

4. M. Obieziurska, A. J. Pacuła, U. Juhas, J. Antosiewicz, J. Ścianowski, *Catalysts* **2018**, *8*, 493-507.

5. A. Laskowska, A. J. Pacuła-Miszewska, A. Długosz-Pokorska, A. Janecka, A. Wojtczak, J. Scianowski, *Materials*, **2022**, *15*, 2068-2082.

6. A. J. Pacuła, M. Obieziurska, J. Ścianowski, K. B. Kaczor, J. Antosiewicz, *Arkivoc*, **2018**, *3*, 144-155.