Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Synthesis of disulfides and diselenides by copper-catalyzed coupling reactions in water

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Supplementary Information

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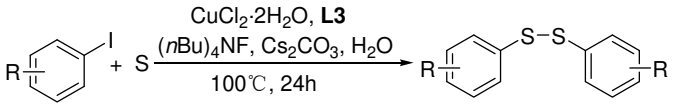
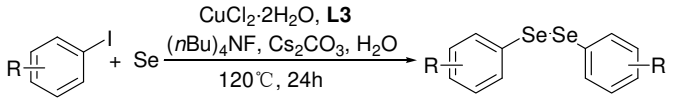
1. General information

All reagents were purchased from commercial suppliers and used without further purification. Analytical thin layer chromatography (TLC) was performed using Merck silica gel GF254 plates. Column chromatography was performed using silica gel (200-300mesh) eluting with ethyl acetate and petroleum ether or with chloroform and methanol. All products were characterized by their NMR. ^{1}H NMR spectra were recorded at 400 MHz and ^{13}C NMR spectra were recorded at 100 MHz (Bruker DPX) with $CDCl_3$ or DMSO-d_6 as solvent. Chemical shifts are reported in ppm using TMS as internal standard. Gas chromatography - mass spectra (GC/MS) were recorded on an Agilent Technologies 6890 N instrument with an Agilent 5973N mass detector (EI) and a HP5-MS 30 m x 0.25 mm capillary apolar column (Stationary phase: 5% diphenyldimethylpolysiloxane film, 0.25 μm). GC/MS method: Initial temperature: 100 °C; Initial time: 1 min; Ramp: about 15°C/min until 250 °C then 20 min.

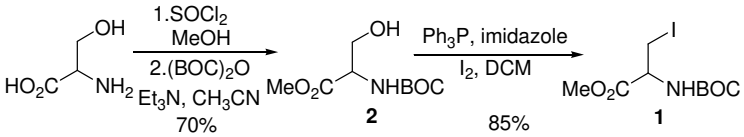
2. General procedure for the catalytic reactions



$CuCl_2$ (0.1 mmol), 1,10-phenanthroline (0.1 mmol), aryl halide (1.0 mmol), Sulfur (3.0 mmol), Cs_2CO_3 (1.0 mmol), (nBu)$_4$NF (0.1 mmol), and water (2 mL) were added to a sealed tube. The reaction mixture was stirred at 100ºC for 24 h and then cooled to room temperature. After the solvent (H_2O) being removed under reduced pressure, the residue was purified by silica-gel column chromatography to afford the corresponding product.



$CuCl_2$ (0.1 mmol), 1,10-phenanthroline (0.1 mmol), aryl halide (1.0 mmol), Selenium (3.0 mmol), Cs_2CO_3 (1.0 mmol), (nBu)$_4$NF (0.1 mmol), and water (2 mL) were added to a sealed tube. The reaction mixture was stirred at 120ºC for 24h and then cooled to room temperature. After the solvent (H_2O) being removed under reduced pressure, the residue was purified by silica-gel column chromatography to afford the corresponding product.

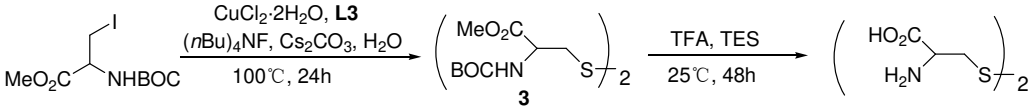


To a suspension of serine (25g, 0.24 mol) in MeOH (240 mL) at 0ºC was added thionyl chloride (17.3 mL, 0.24 mol) slowly, the suspension starts to dissolve during the addition, and when addition is complete, the mixture is brought to r.t. and stirred 1 hr. The stirring is then stopped, and the mixture was let stand overnight. The MeOH was then removed in vacuo, and the resulting solid was filtered, and washed with ether. The solid was then recrystallized from MeOH/ether to yield 25.4 g (70%) of a white crystallize solid.

To a solution of Boc-anhydride (14 g, 64 mmol) in acetonitrile (200 mL) was added Lserine methyl

ester hydrochloride salt (9.98 g, 64 mmol) and triethylamine (26.8 mL, 190 mmol) in one portion. The reaction was stirred for 6 hrs, DCM (1 L) was added, and extracted with 1 N HCl (700 mL), sat. sodium bicarbonate (100 mL). The organic layer was dried ($MgSO_4$), and the solvent was removed in vacuo, and placed under high vacuum overnight to remove the remainder of the solvent to yield Boc-serine-methylester (14 g, 99%) as a thick oil.

To a solution of triphenylphosphine (20.9 g, 79.8 mmol) and imidazole (5.4 g, 79.8 mmol) in DCM (300 mL) at 0ºC was added I_2 (20.2 g, 79.8 mmol) in three portions. The solution was warmed to r.t. and stirred for 10 min, and re-cooled to 0ºC. **2** (14 g, 64 mmol) was then added in DCM (75 mL, washed with 10 mL) dropwise. The solution was stirred at this temperature for 1 hr and r.t. for 1.5 hr. The reaction mixture was then filtered through silica gel using 50/50 ether/pet. ether as eluent. The solvent was then removed in vacuo. Ether was then added to crash out the phosphine oxide, and the the mixture was filtered through celite with ether as the eluent. The solvent was again removed in vacuo, and the residue was purified on silica (10-15% ether/PE) to yield **1** (17.5 g, 84%) as an oil which solidified in the freezer.

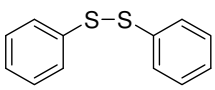


$CuCl_2{\cdot}2H_2O$ (0.1 mmol), 1,10-phenanthroline (0.1 mmol), **1** (1.0 mmol), Sulfur (3.0 mmol), Cs_2CO_3 (1.0 mmol), (nBu)$_4$NF (0.1 mmol), and water (2 mL) were added to a sealed tube. The reaction mixture was stirred at 100ºC for 24h and then cooled to room temperature. After the solvent (H_2O) being removed under reduced pressure, the residue was purified by silica-gel column chromatography to afford the corresponding product.

To a solution of **3** (1 mmol) in DMF (30 mL) was added p-chloroaniline (1.2 mmol), 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide (EDC, 1 mmol), HOBt (1.2 mmol), and DIPEA (2.4 mmol). The reaction mixture was stirred at RT for 2 h. After reaction was complete, 30 mL of 1M HCl was added, and the mixture was extracted with EtOAc. The combined organic layers were washed with brine, dried over $MgSO_4$, and filtered. EtOAc was removed under vacuum, yielding cystine, which was further purified by column chromatography (SiO_2/3:7 ethyl acetate/hexane) to give a white solid.

3. Experimental procedures and characterization data

1,2-diphenyldisulfane[1]



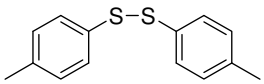
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.25-7.55 (m, 10H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 155.2, 129.7, 120.9, 115.4.

MS (EI, m/z): 218 [M+].

1,2-di-*p*-tolydisulfane[1]



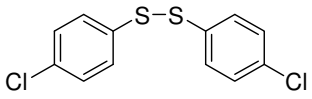
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.99 (d, 4H, *J*=8Hz), 6.72(d, 4H, *J*=8Hz), 2.24(s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 152.8, 130.2, 130.1, 115.3, 20.5.

MS (EI, m/z): 2**46** [M+].

1,2-bis(4-chlorophenyl)disulfane[1]



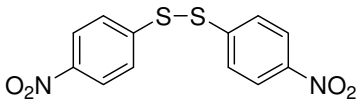
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.01(d, 4H, *J*=3.2Hz), 7.65(d, 4H, *J*=2.8Hz).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 145.0, 129.1, 123.2, 116.3.

MS (EI, m/z): 2**87** [M+].

1,2-bis(4-nitrophenyl)disulfane[1]



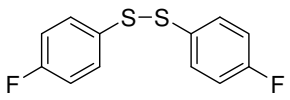
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, **DMSO-d_6)** δ= 7.65 (d, 4H, *J*=8Hz), 6.92 (d, 4H, *J*=8Hz).

^{13}C NMR (100 MHz, $CDCl_3$) δ= 152.5, 139.1, 126.4, 113.4.

MS (EI, m/z): 308 [M+].

1,2-bis(4-fluorophenyl)disulfane[2]



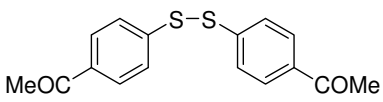
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ =6.88-6.82(m, 4H), 6.64-6.60(m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 157.6, 155.3, 142.4, 116.1, 116.0, 115.8, 115.6.

MS (EI, m/z): 254 [M+].

1,2-bis(4-acetylphenyl)disulfane[3]



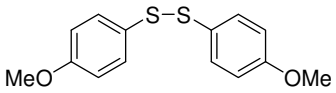
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.91(d, 4H, J=8.4Hz), 7.55(d, 4H, J=8.0Hz), 2.56(s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 196.9, 142.3, 135.8, 129.1, 126.1, 26.6.

MS (EI, m/z): 302 [M+].

1,2-bis(4-methoxyphenyl)disulfane[1]



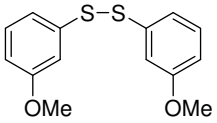
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.59(d, 4H, *J*=8.8Hz), 6.71(d, 4H, *J*=9.2Hz), 3.80 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 159.5, 138.2, 116.4, 82.8, 55.37.

MS (EI, m/z): 278 [M+].

1,2-bis(3-methoxyphenyl)disulfane[2]

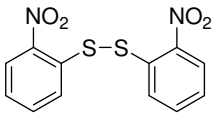


^{1}H NMR (400 MHz, $CDCl_3$) δ =7.55-6.82(m, 8H), 3.83 (s, 6H)..

^{13}C NMR (100 MHz, $CDCl_3$) δ = 160.4, 134.2, 133.8, 132.7, 127.4, 114.7, 55.4.

MS (EI, m/z): 2**78** [M+].

1,2-bis(2-nitrophenyl)disulfane[4]



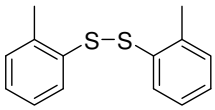
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, CDCl3) δ = 8.39(d, 2H, J=8.0Hz), 7.76-7.87 (m, 4H), 7.58 (d, J=7.6Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 146.0, 135.9, 133.8, 128.3, 127.4, 127.1.

MS (EI, m/z): 308 [M+].

1,2-dio-tolydisulfane[2]



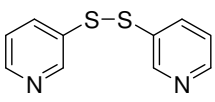
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.26-7.08 (m, 8H), 2.41 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 135.3, 132.2, 129.1, 128.4, 123.1, 120.6, 46.7.

MS (EI, m/z): 2**46** [M+].

3-(2-(pyridine-3-yl)disulfanyl)pyridine[5]



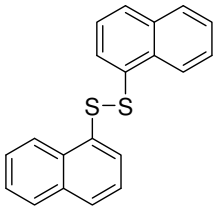
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, CDCl3) δ = 8.71-8.56(m, 4H), 7.73-7.71(m, 2H), 7.20 (s, 2H).

^{13}C NMR (100 MHz, CDCl3) δ = 153.0, 148.7, 140.6, 127.8, 124.8.

MS (EI, m/z): 220 [M+].

1,2-di(naphthalen-1-yl)disulfane[4]



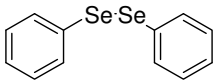
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow soild.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.36((d, 2H, *J*=8.4Hz), 7.86-7.91(m, 6H), 7.36-7.68(m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 134.7, 132.1, 130.0, 128.4, 128.1, 127.5, 127.2, 126.8, 126.3, 123.0.

MS (EI, m/z): 3**18** [M+].

1,2-diphenyldiselane[6]



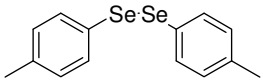
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow soild.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.35-7.75(m, 10H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 133.1, 131.7, 129.4, 127.9.

MS (EI, m/z): 312 [M+].

1,2 dip-tolydiselane[6]



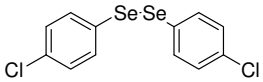
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.53(d, 4H, *J*=7.6Hz), 7.11(d, 4H, *J*=7.6Hz), 2.37(s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 138.0, 133.4, 132.3, 129.4, 21.1.

MS (EI, m/z): 340 [M+].

1,2-bis(4-chlorophenyl)diselane[6]



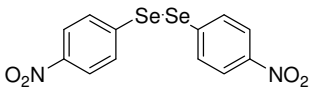
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ =7.18 (d, 4H, *J*=8Hz), 6.76(d, 4H, *J*=8Hz).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 153.7, 129.6, 125.8, 116.7.

MS (EI, m/z):381 [M+].

1,2-bis(4-nitrophenyl)diselane[7]



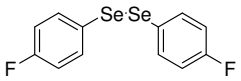
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ =7.33 (d, 4H, J=8Hz), 6.72(d, 4H, J=8Hz).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 154.2, 132.5, 117.2, 113.1.

MS (EI, m/z): 402 [M+].

1,2-bis(4-fluorophenyl)diselane[8]



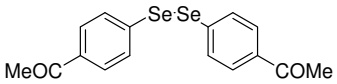
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ =δ 6.90-6.94 (m, 4H), 6.75-6.79 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 158.6, 156.2, 150.9, 116.4, 116.3, 116.2, 116.0.

MS (EI, m/z): 348 [M+].

1,2-bis(4- acetylphenyl)diselane[9]



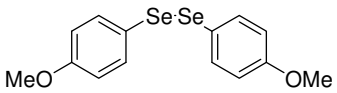
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.79(d, 4H, *J*=8Hz), 7.55(d, 4H, *J*=8Hz), 2.61(s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 197.3, 136.2, 132.7, 130.2, 129.2, 29.7.

MS (EI, m/z): 396 [M+].

1,2-bis(4-methoxyphenyl)diselane[6]



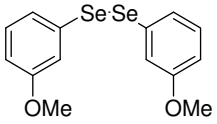
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.54(d, 4H, *J*=8.4Hz), 6.84(d, 4H, *J*=8.4Hz), 3.83(s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 160.1, 135.5, 122.0, 114.8, 55.3.

MS (EI, m/z): 372 [M+].

1,2-bis(3-methoxyphenyl)diselane[6]



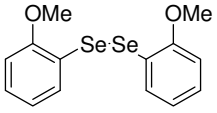
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.13-7.19(m, 6H), 6.89(d, 2H, *J*=8Hz), 3.82(s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 160.4, 130.6, 123.8, 122.9, 117.2, 113.1, 55.4.

MS (EI, m/z): 372 [M+].

1,2-bis(2-methoxyphenyl)diselane[6]



Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

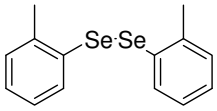
^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.57(d, 2H, *J*=8Hz), 7.28-7.32(m, 2H), 6.92(d, 2H, *J*=8Hz), 6.86(m, 2H)), 3.91(s, 6H)). ^{13}C NMR($CDCl_3$, 100 MHz) δ 155.8, 133.4, 128.6, 121.8, 1120, 111.7, 56.2. MS (EI, *m/z*): 372 [M^+].

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.57(d, 2H, *J*=8Hz), 7.28-7.32(m, 2H), 6.92(d, 2H, *J*=8Hz), 6.86(m, 2H)), 3.91(s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 155.8, 133.4, 128.6, 121.8, 1120, 111.7, 56.2.

MS (EI, m/z): 372 [M+].

1,2-dio-tolydiselane[6]



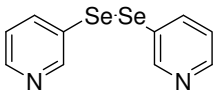
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ =8.41-7.57(m, 8H), 3.35(s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 138.7, 133.2, 131.2, 131,1, 130.0, 128.6, 21.4.

MS (EI, m/z): 340 [M+].

3-(2-(pyridine-3-yl)diselanyl)pyridine[6]



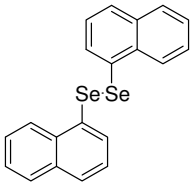
Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.51-7.50(m, 10H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 152.5, 148.1, 140.2, 133.6, 129.6.

MS (EI, m/z): 314 [M+].

1,2-di(naphthalen-1-yl)diselane[9]



Purification by flash chromatography (petroleum ether/ethyl acetate 30:1). Yellow soild.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.32-8.34(m, 2H), 7.84-7.90(m, 6H), 7.35-7.68(m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 134.7, 132.1, 130.0, 128.4, 128.0, 127.4, 127.2, 126.8, 126.3, 123.0.

MS (EI, m/z): 412 [M+].

4. References

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5. Ms, ^{1}H NMR and ^{13}C NMR spectra for the products

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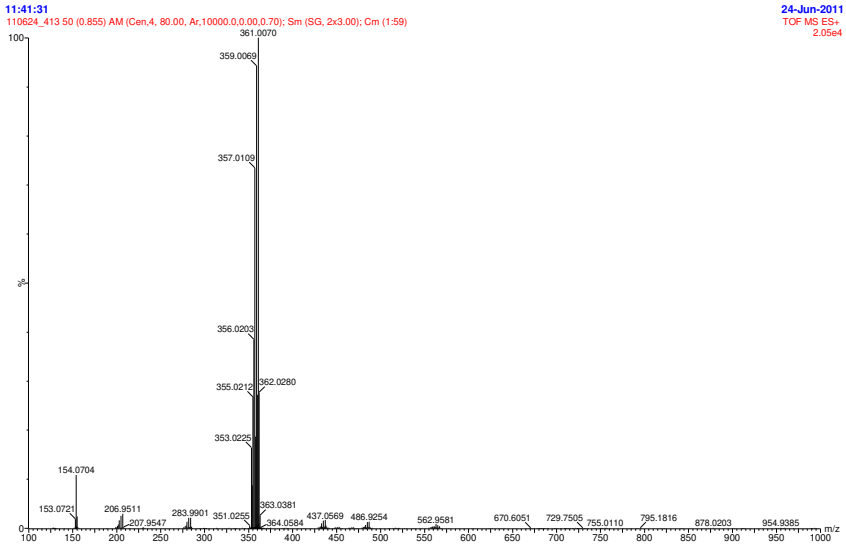
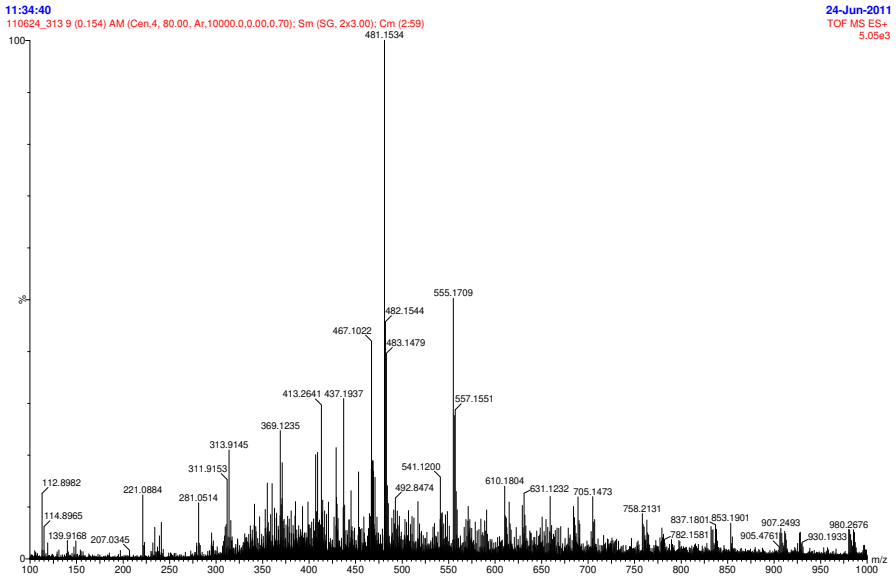


1,2-diphenyldiselane



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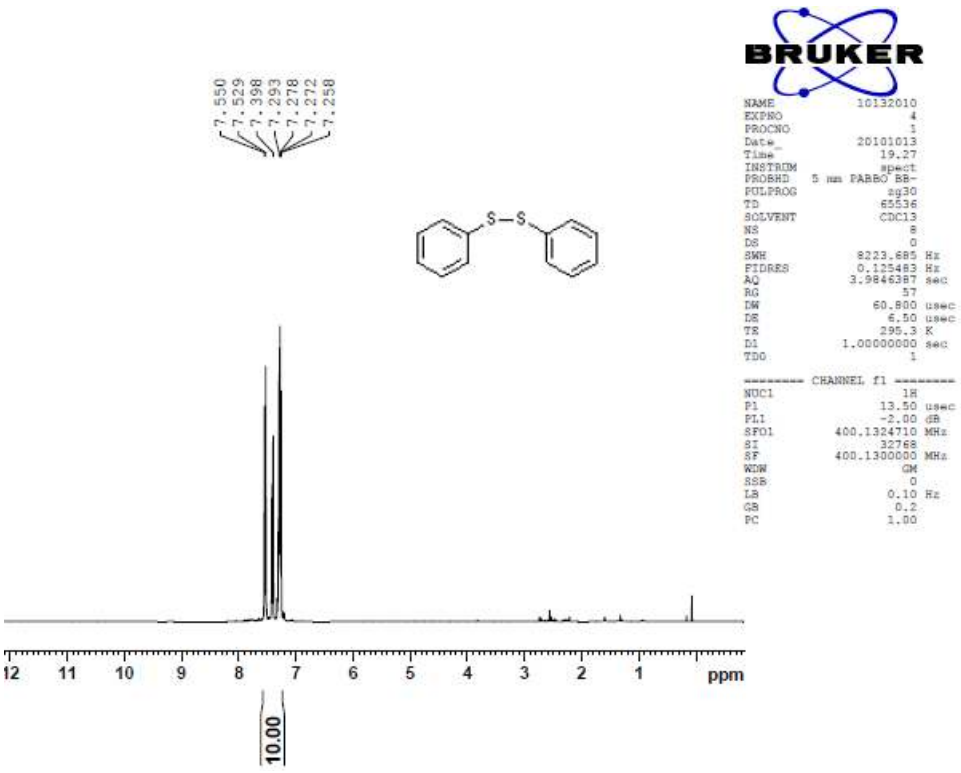
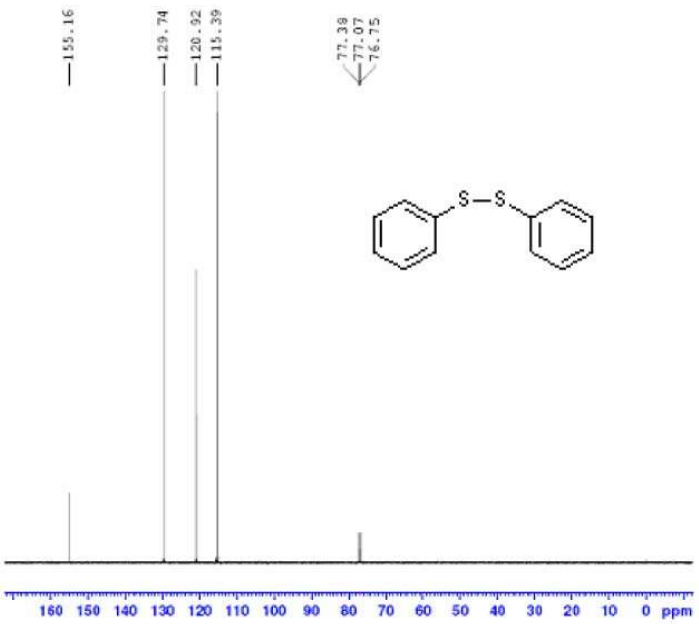
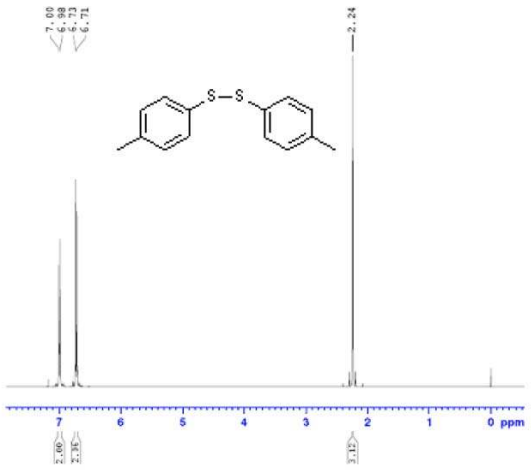
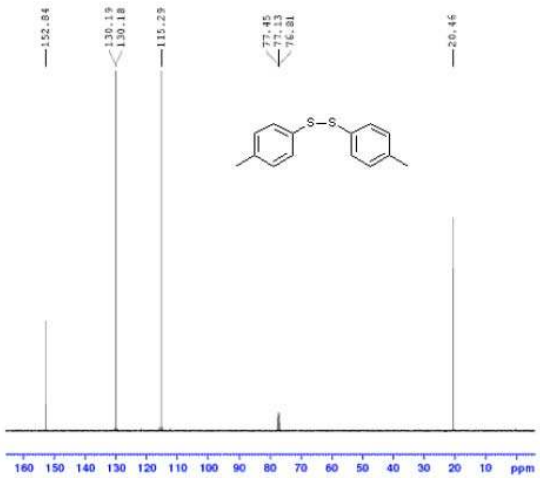
1,2-diphenyldisulfane





1,2-di-*p*-tolydisulfane





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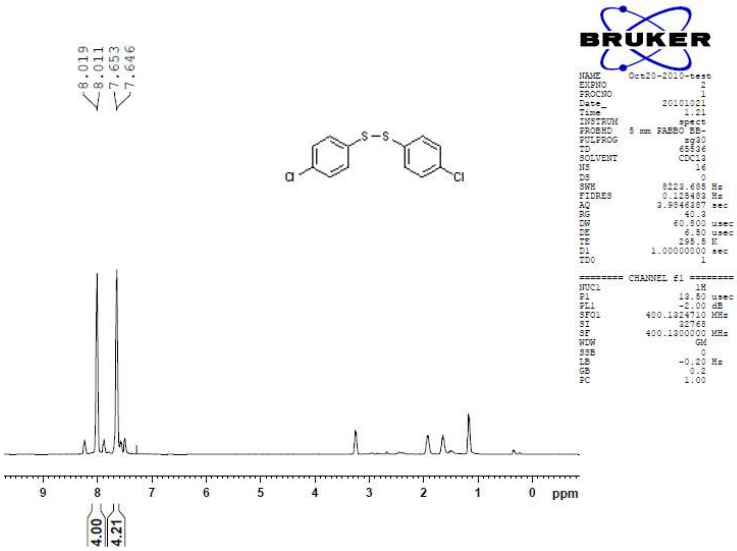
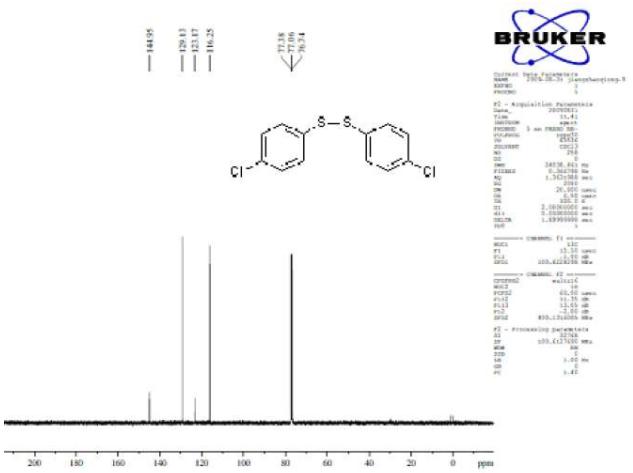
1,2-bis(4-chlorophenyl)disulfane





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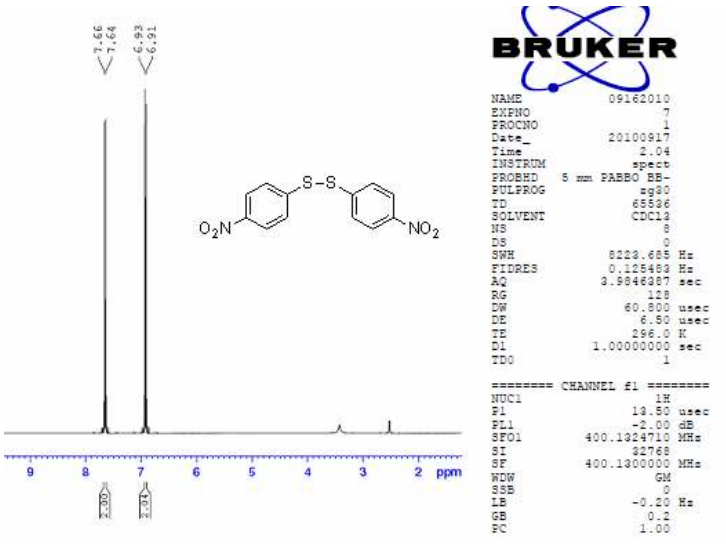
1,2-bis(4-nitrophenyl)disulfane

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D1                1.00000000 sec
TD0                        1

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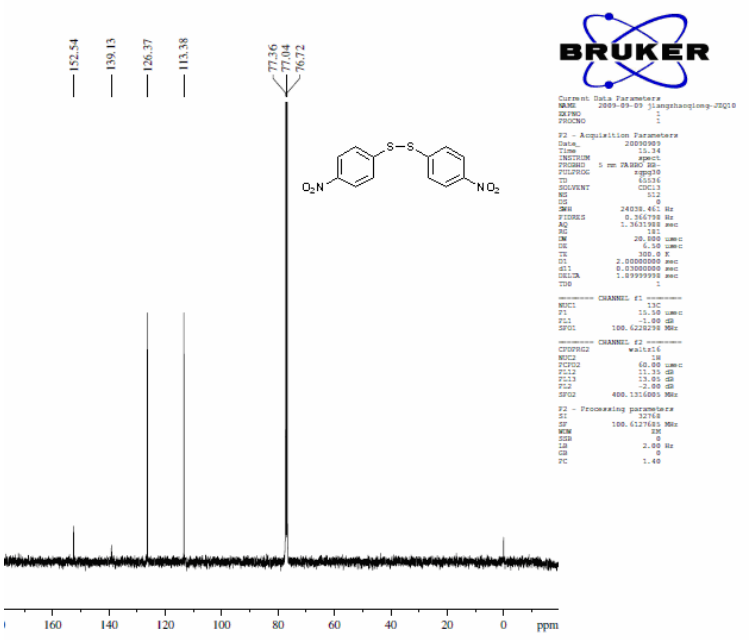

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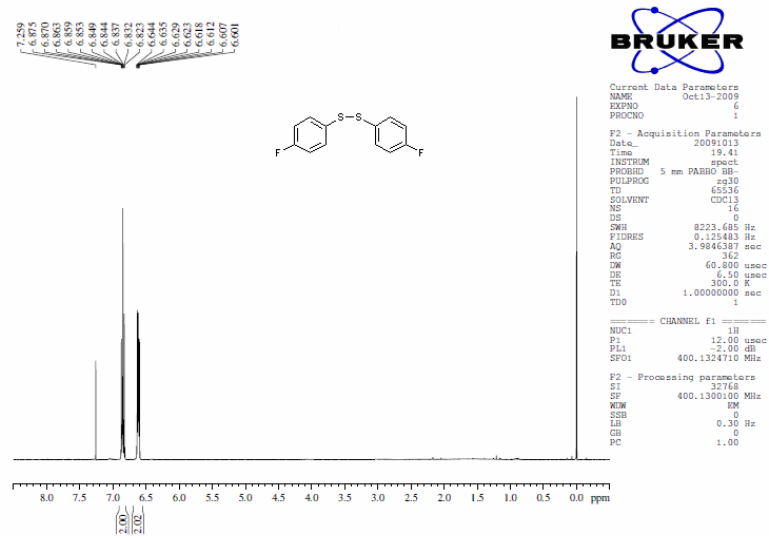

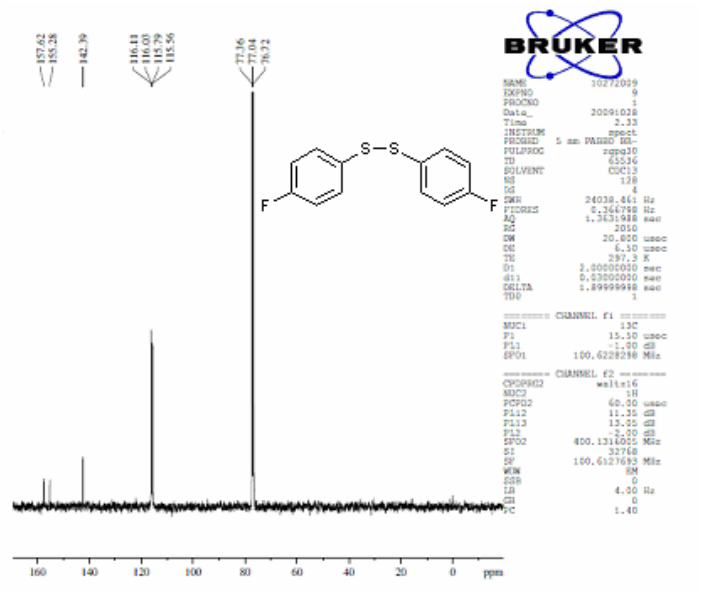

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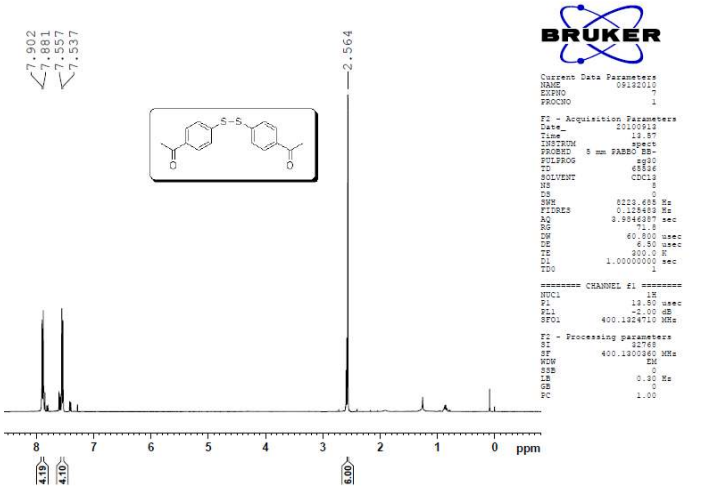
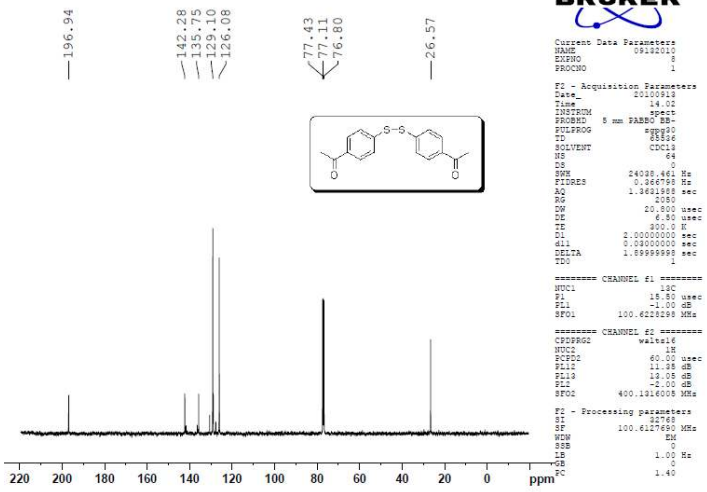
1,2-bis(4-acetylphenyl)disulfane





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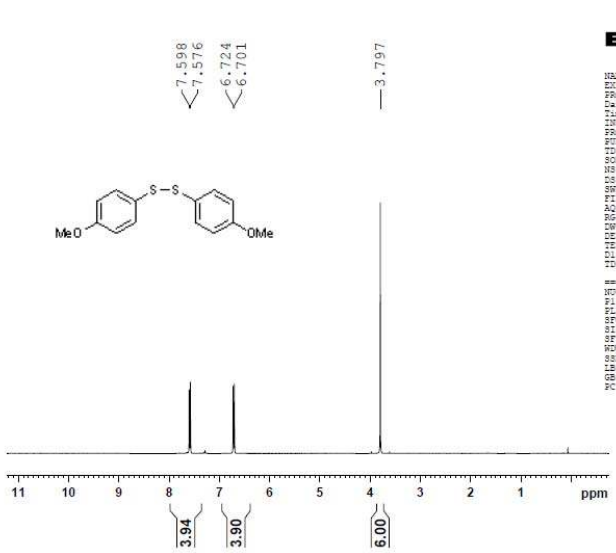
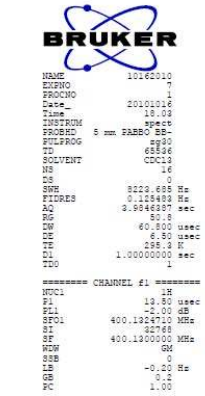
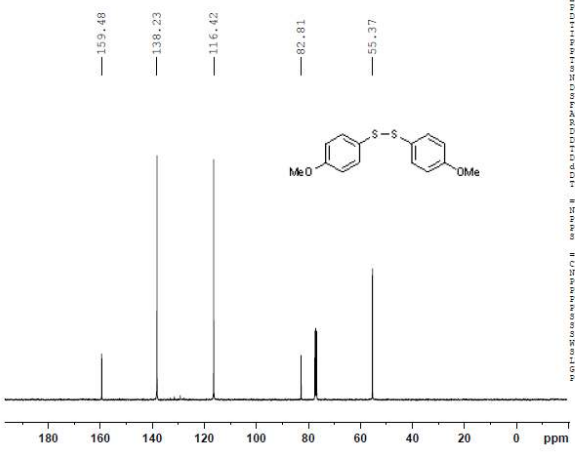
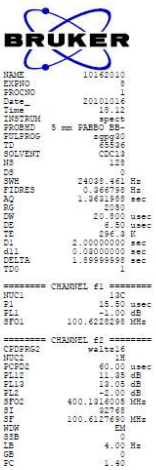
1,2-bis(4-methoxyphenyl)disulfane









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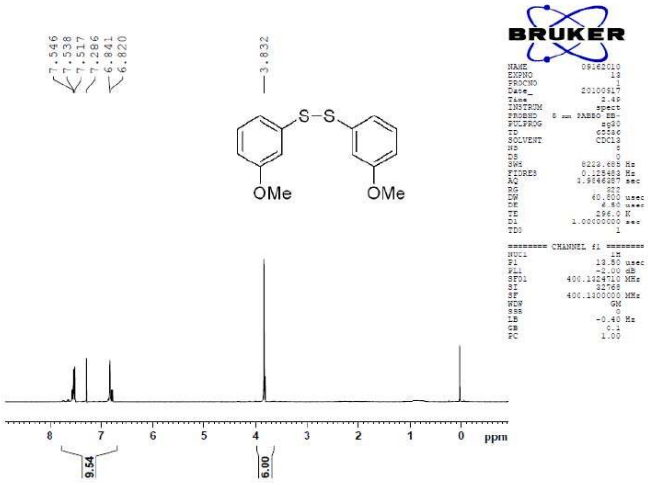
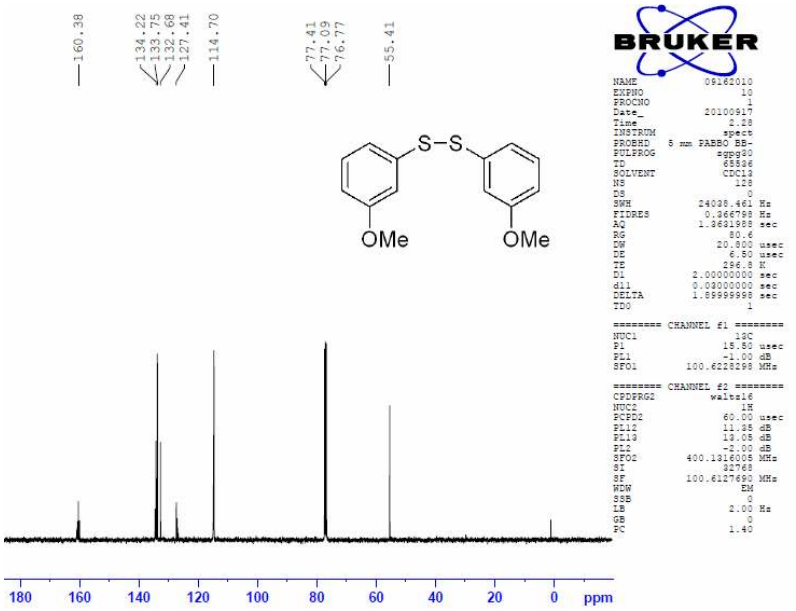
1,2-bis(3-methoxyphenyl)disulfane





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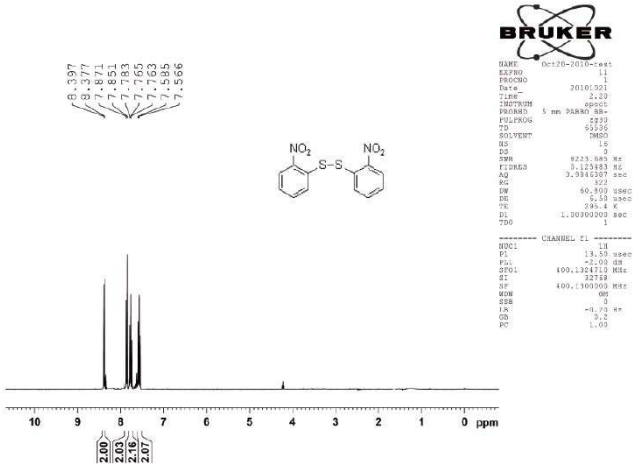

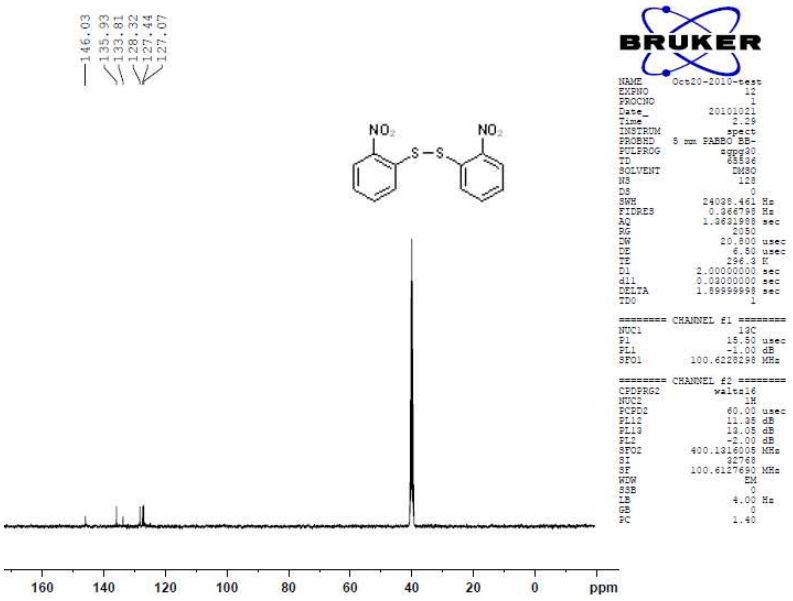

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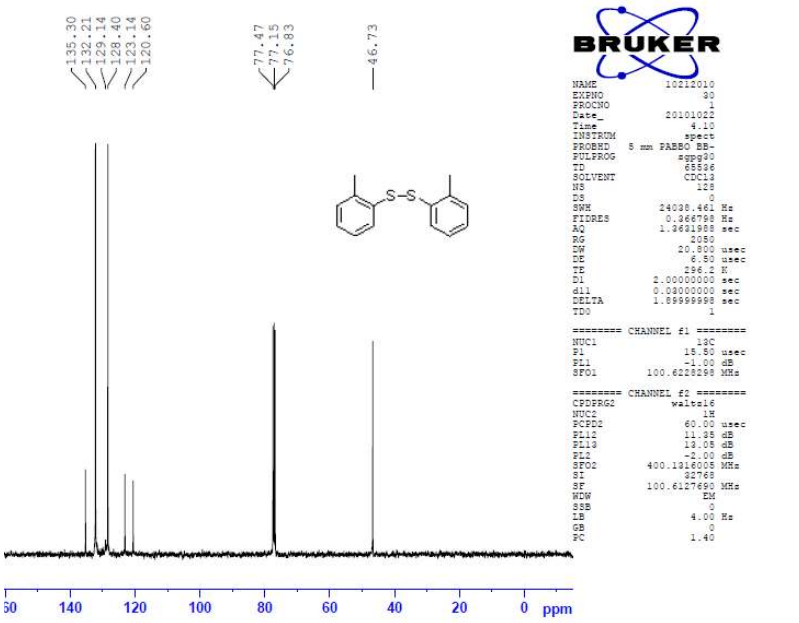
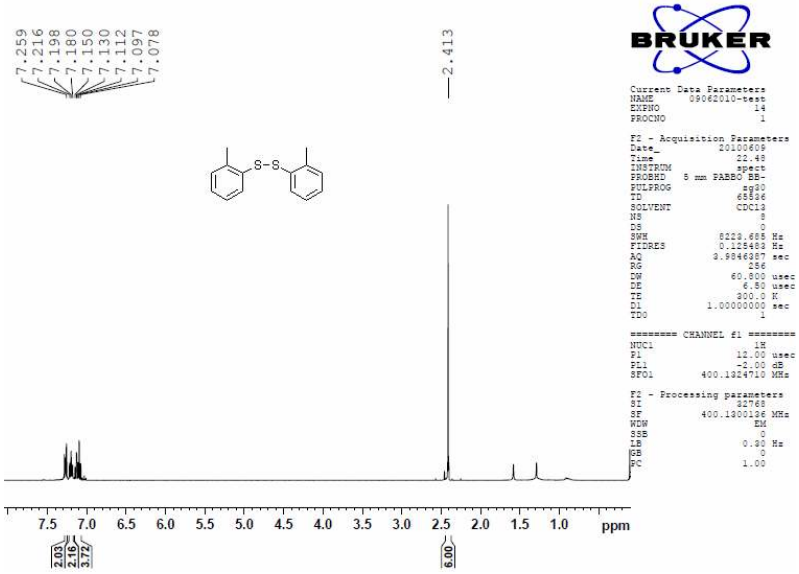
1,2-di-*o*-tolydisulfane





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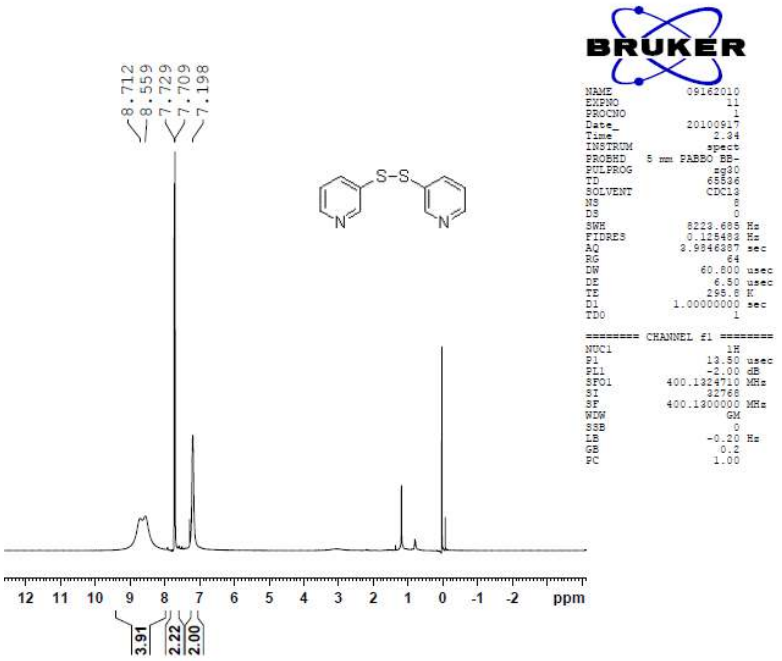
3-(2-(pyridine-3-yl)disulfanyl)pyridine

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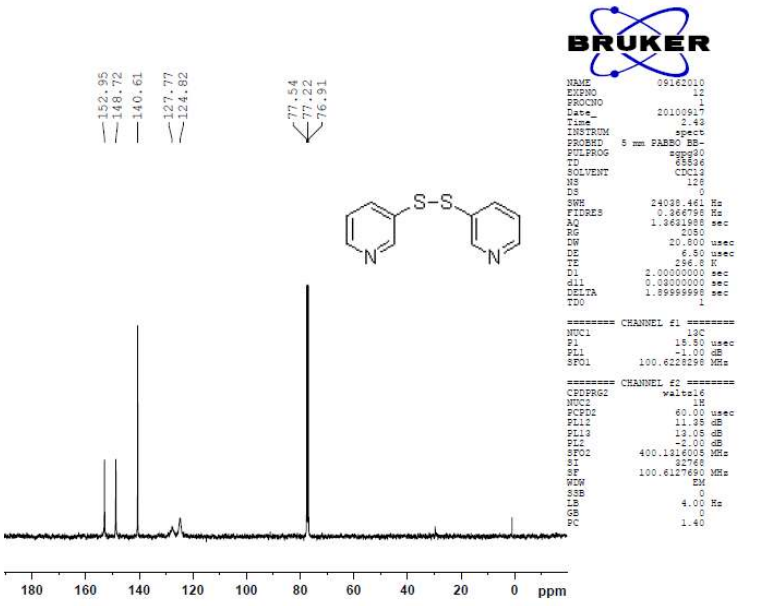


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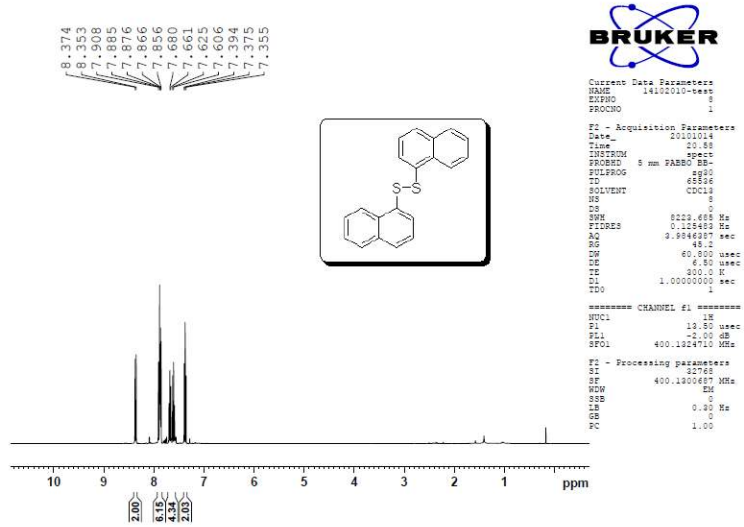
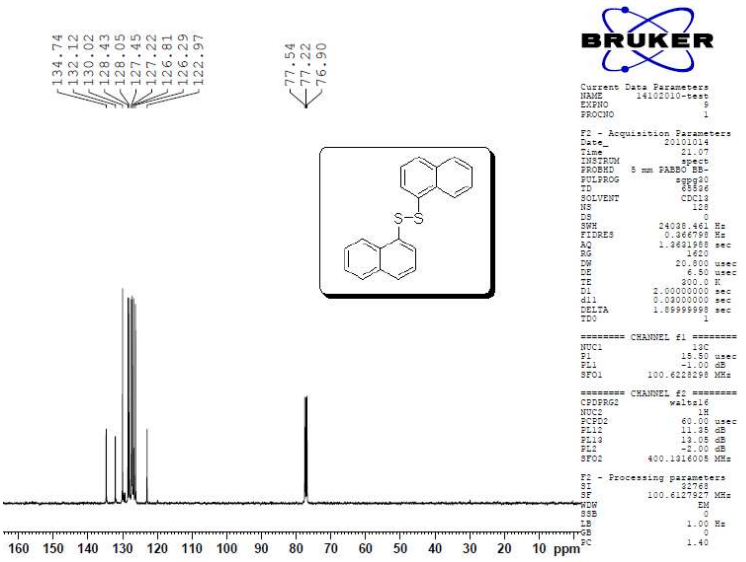
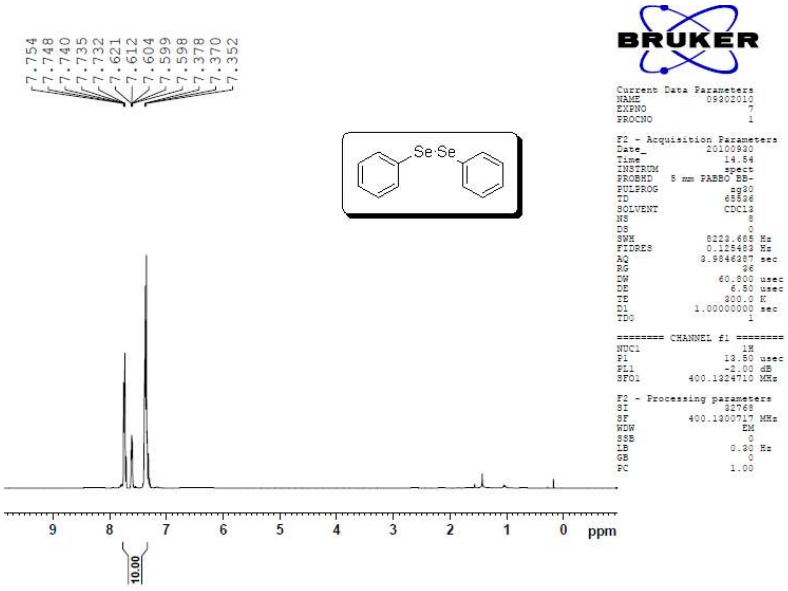
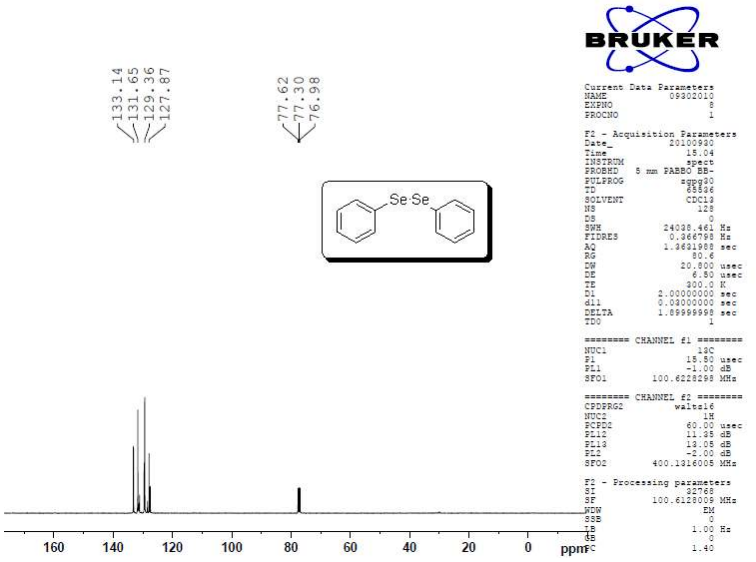
1,2-di(naphthalen-1-yl)disulfane





1,2-diphenyldiselane





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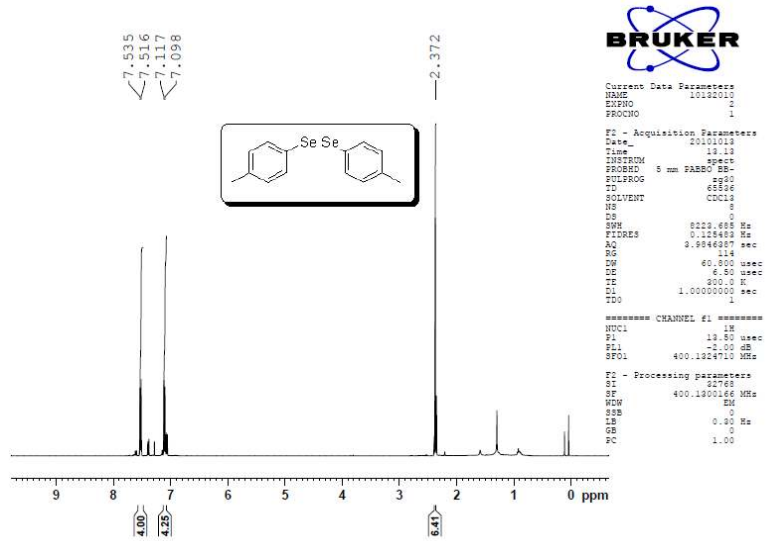
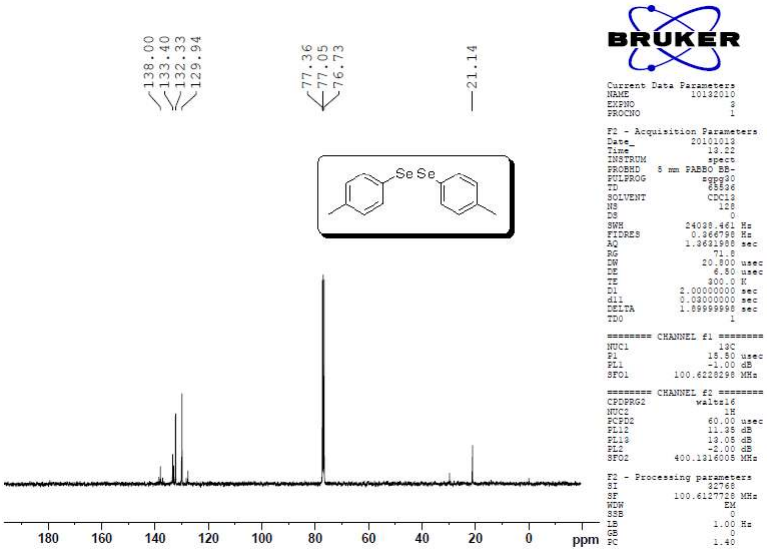
1,2-di-*p*-tolydiselane





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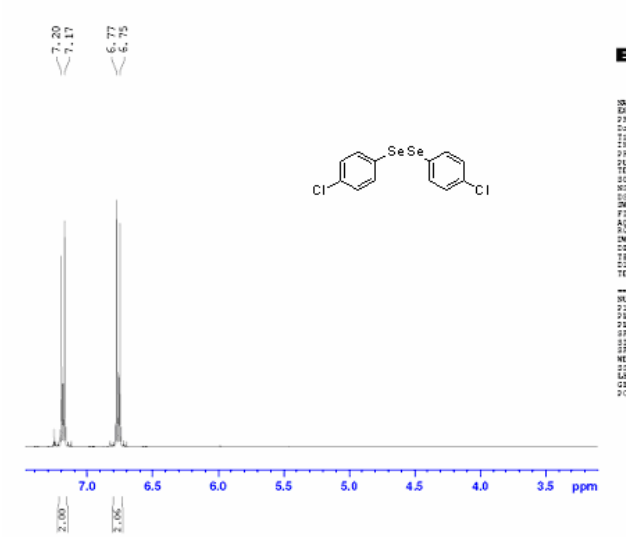
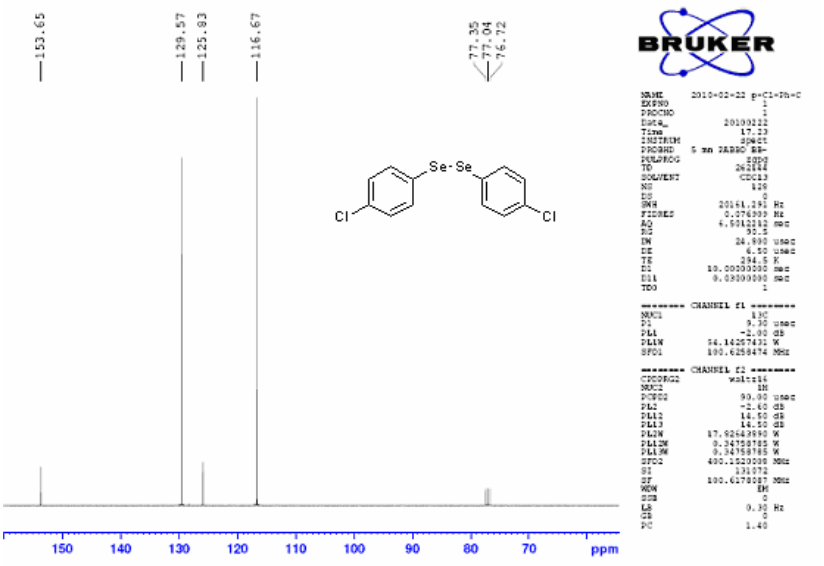
1,2-bis(4-chlorophenyl)diselane





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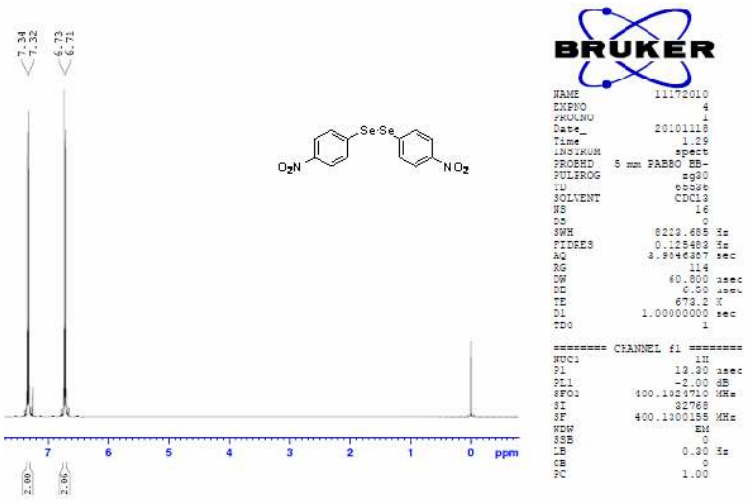
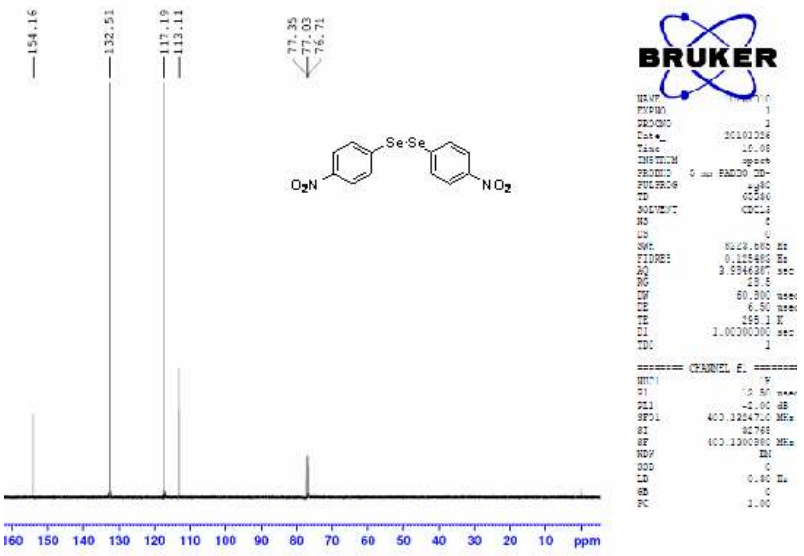
1,2-bis(4-nitrophenyl)diselane





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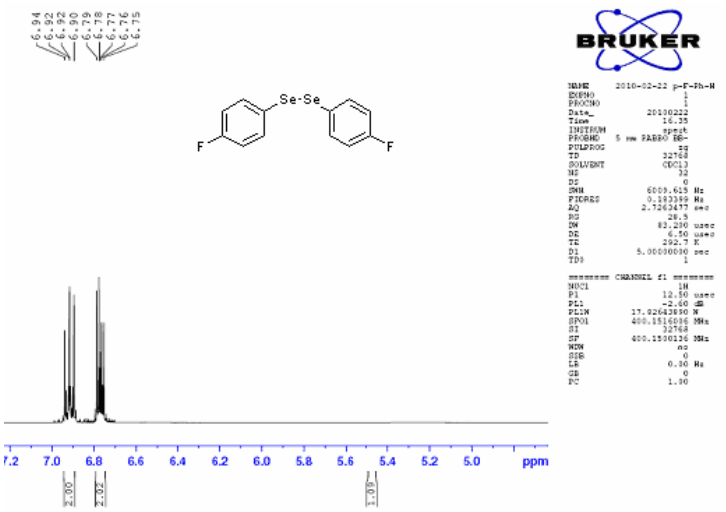
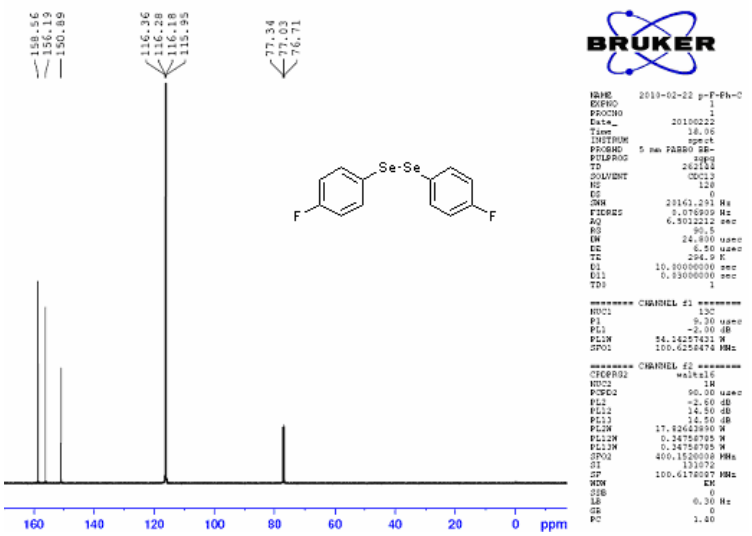
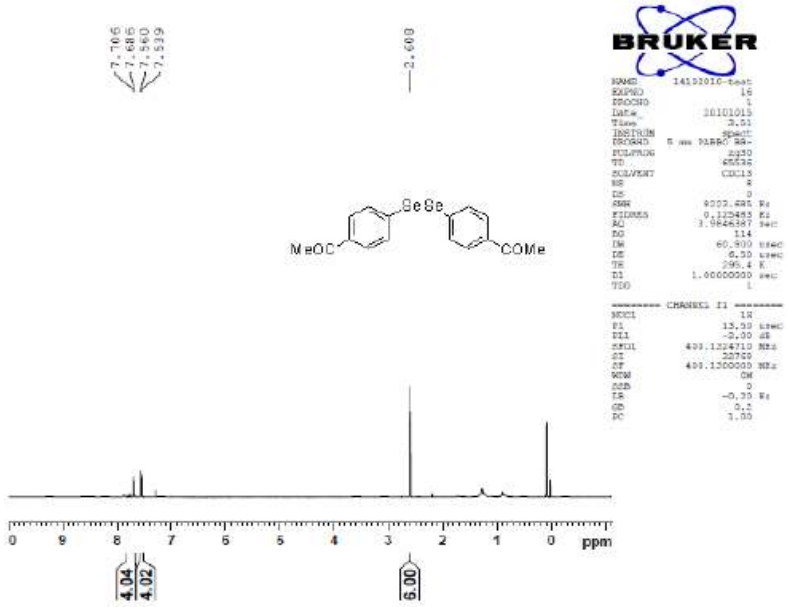
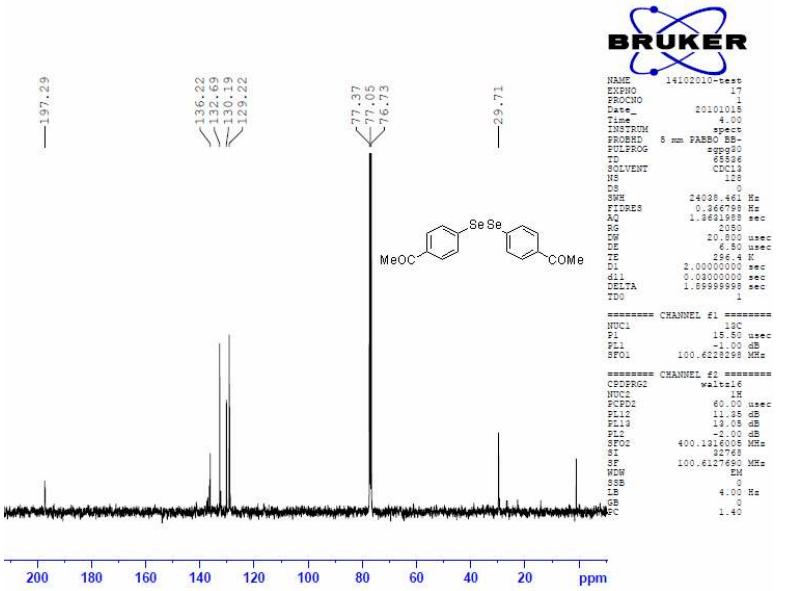
1,2-bis(4-fluorophenyl)diselane





1,2-bis(4-acetylphenyl)diselane





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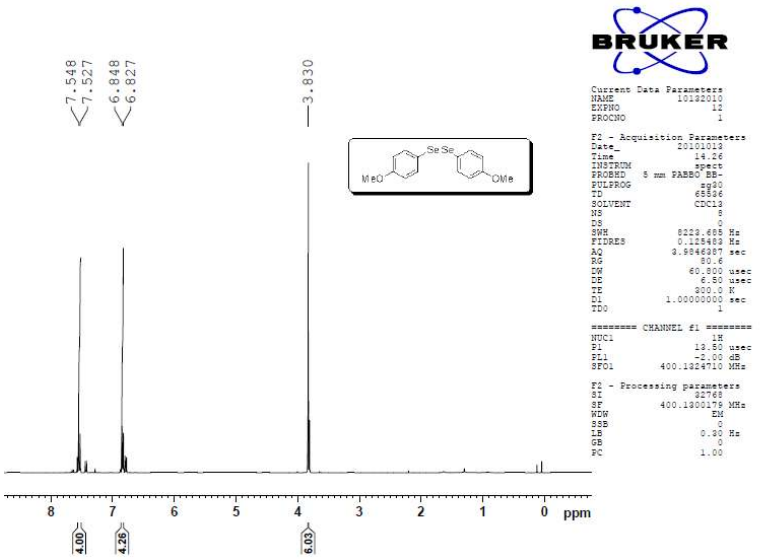
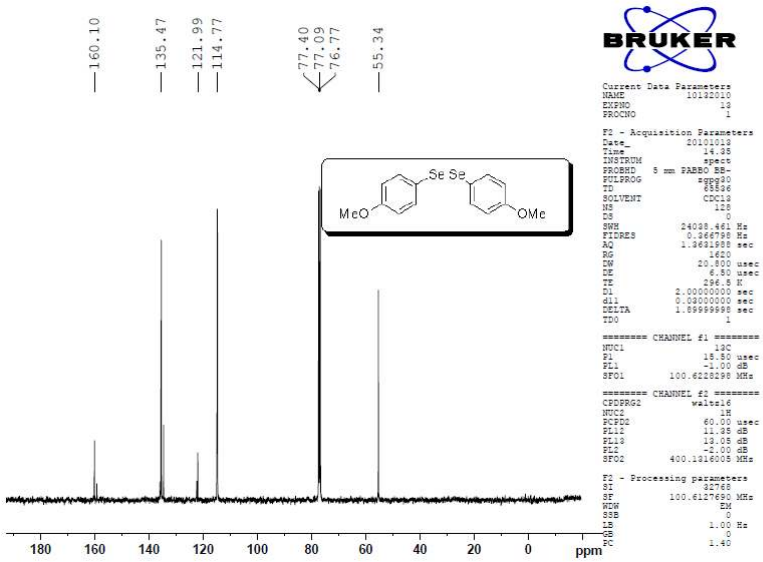
1,2-bis(4-methoxyphenyl)diselane





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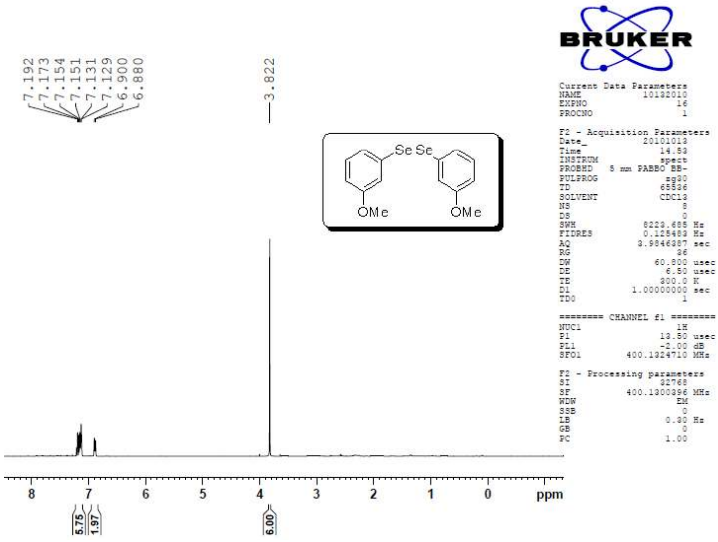
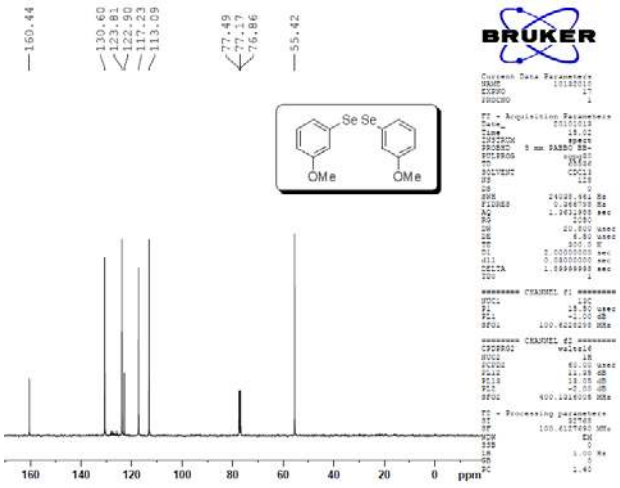
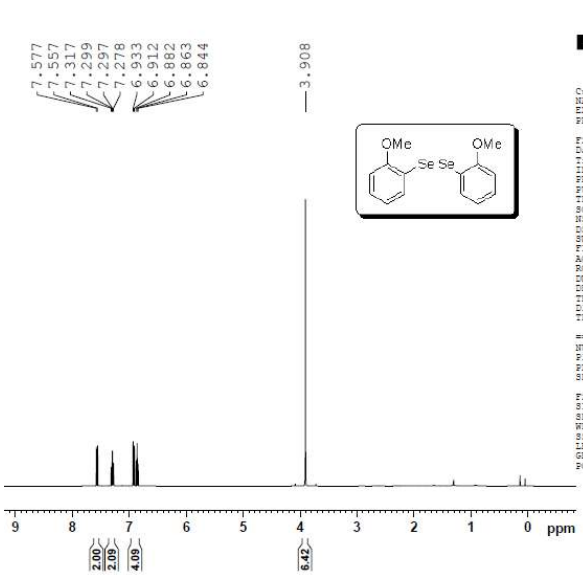
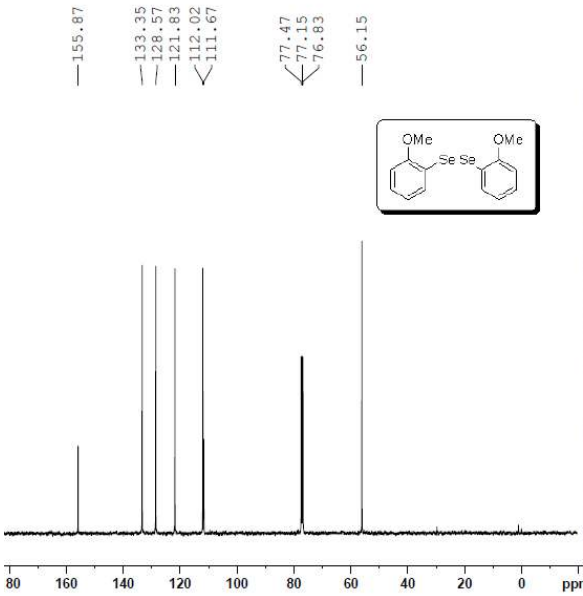
1,2-bis(3-methoxyphenyl)diselane





1,2-bis(2-methoxyphenyl)diselane





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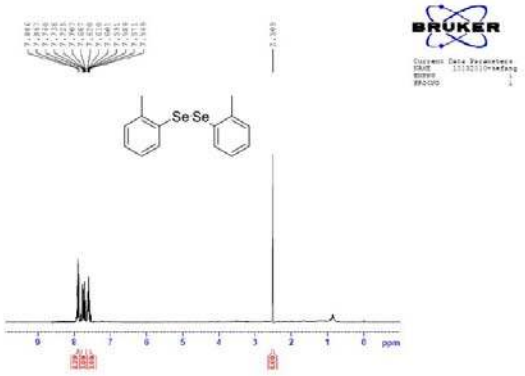
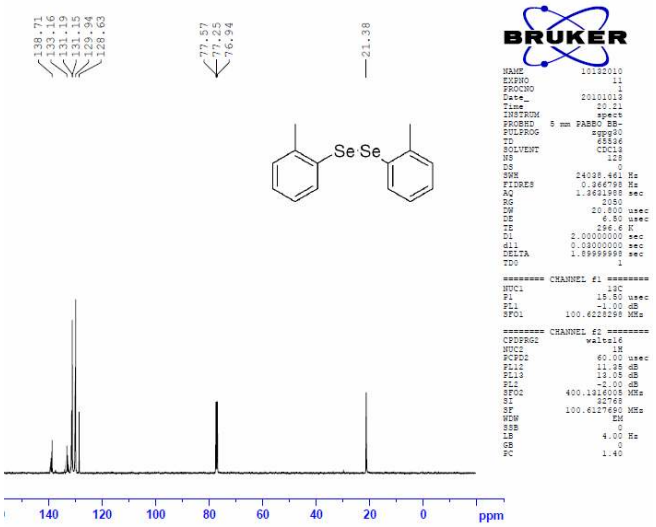
1,2-di-*o*-tolydiselane





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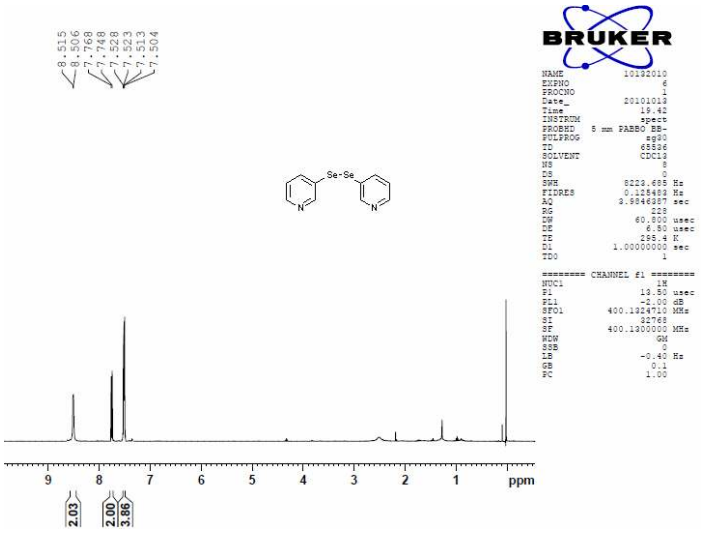
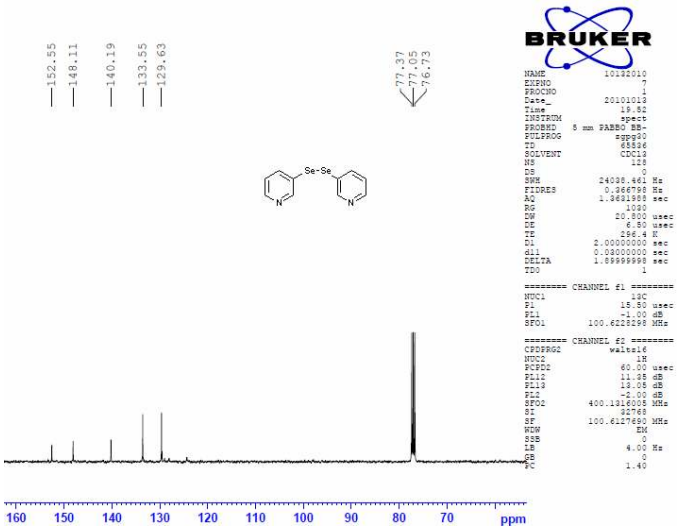
3-(2-(pyridine-3-yl)diselanyl)pyridine





1,2-di(naphthalen-1-yl)diselane