

Supplementary Information

***In situ synthesis of CuO nanoparticles over functionalized mesoporous silica
and their application in catalytic syntheses of symmetrical diselenides***

Trisha Das,^a Rana Chatterjee,^b Adinath Majee,^b Hiroshi Uyama,^c David Morgan^d and Mahasweta Nandi*^a

^a*Integrated Science Education and Research Centre, Siksha Bhavana, Visva-Bharati University,
Santiniketan 731 235, India*

Email: mahasweta.nandi@visva-bharati.ac.in

^b*Department of Chemistry, Siksha Bhavana, Visva-Bharati University, Santiniketan 731235, India*

^c*Department of Applied Chemistry, Graduate School of Engineering, Osaka University, 2-1 Yamadaoka,
Suita, Osaka, 565-0871, Japan*

^d*Cardiff Catalysis Institute, School of Chemistry, Cardiff University, Park Place, Cardiff CF10 3AT*

1,2-diphenyldiselenane (3a)¹: (243 mg, Yield: 78%); ¹H NMR (CDCl_3 , 400 MHz): δ (ppm) 7.65-7.63 (m, 4H), 7.32-7.27 (m, 6H); ¹³C NMR (CDCl_3 , 100 MHz): δ (ppm) 131.6, 131.0, 129.3, 127.8; ⁷⁷Se NMR (CDCl_3 , 76.28 MHz): δ (ppm) 463.09.

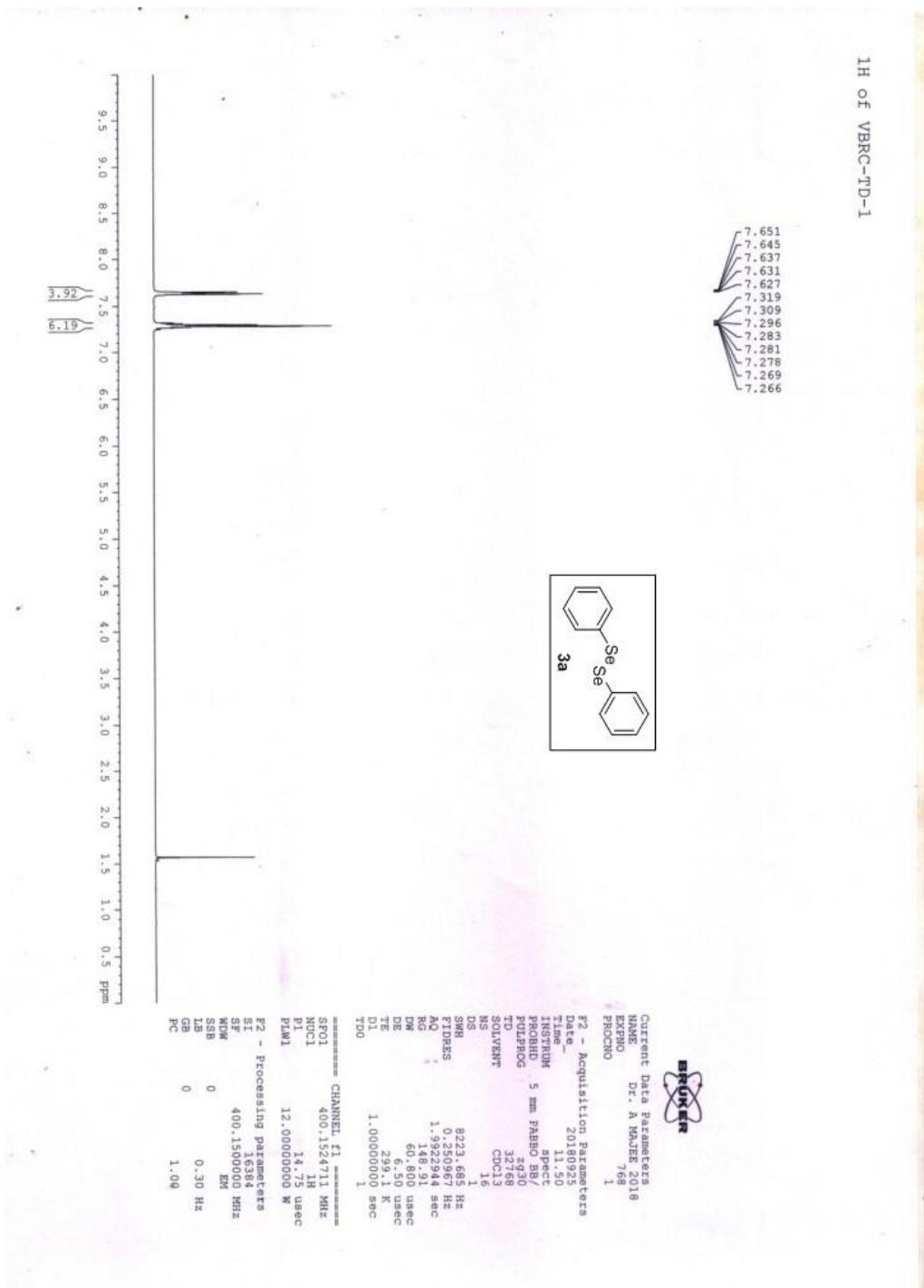
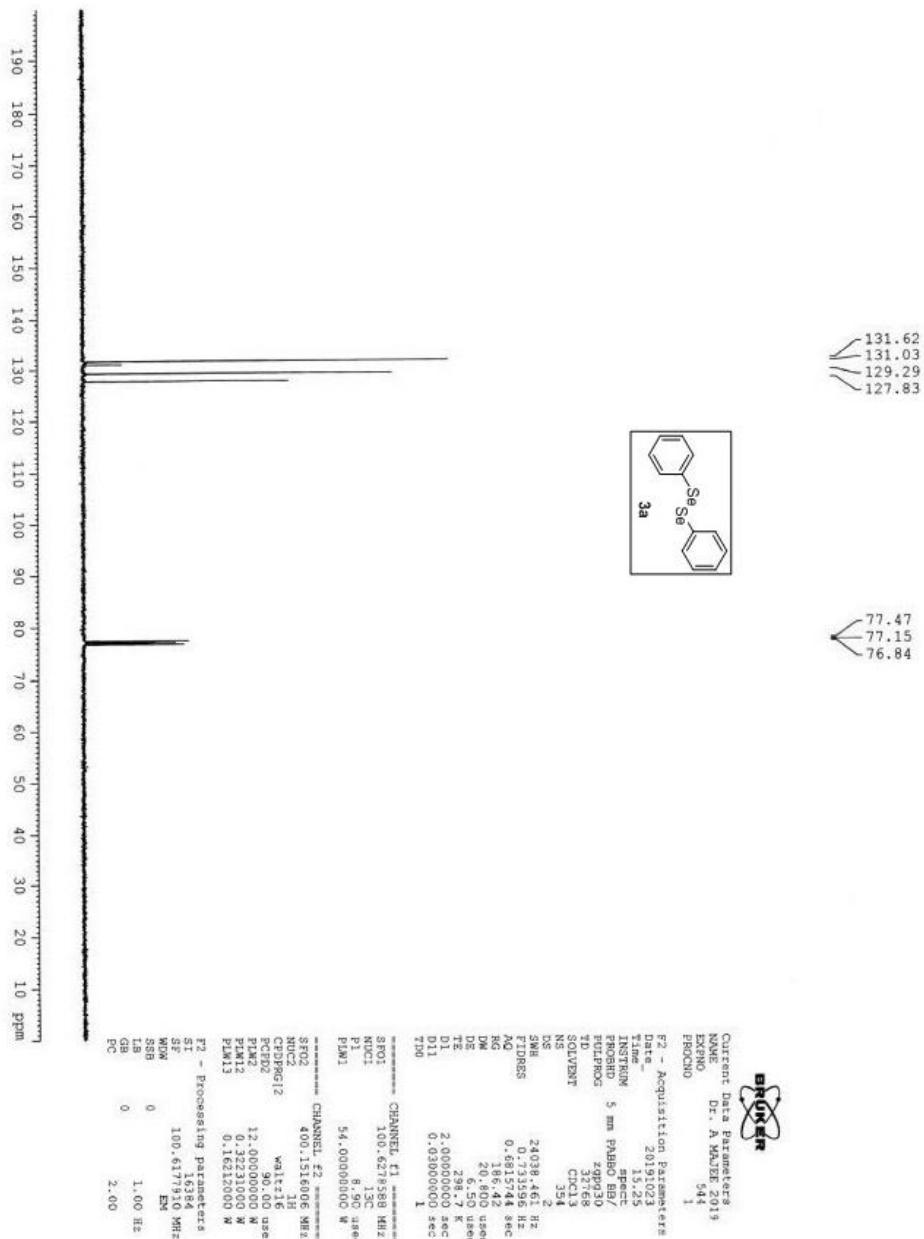
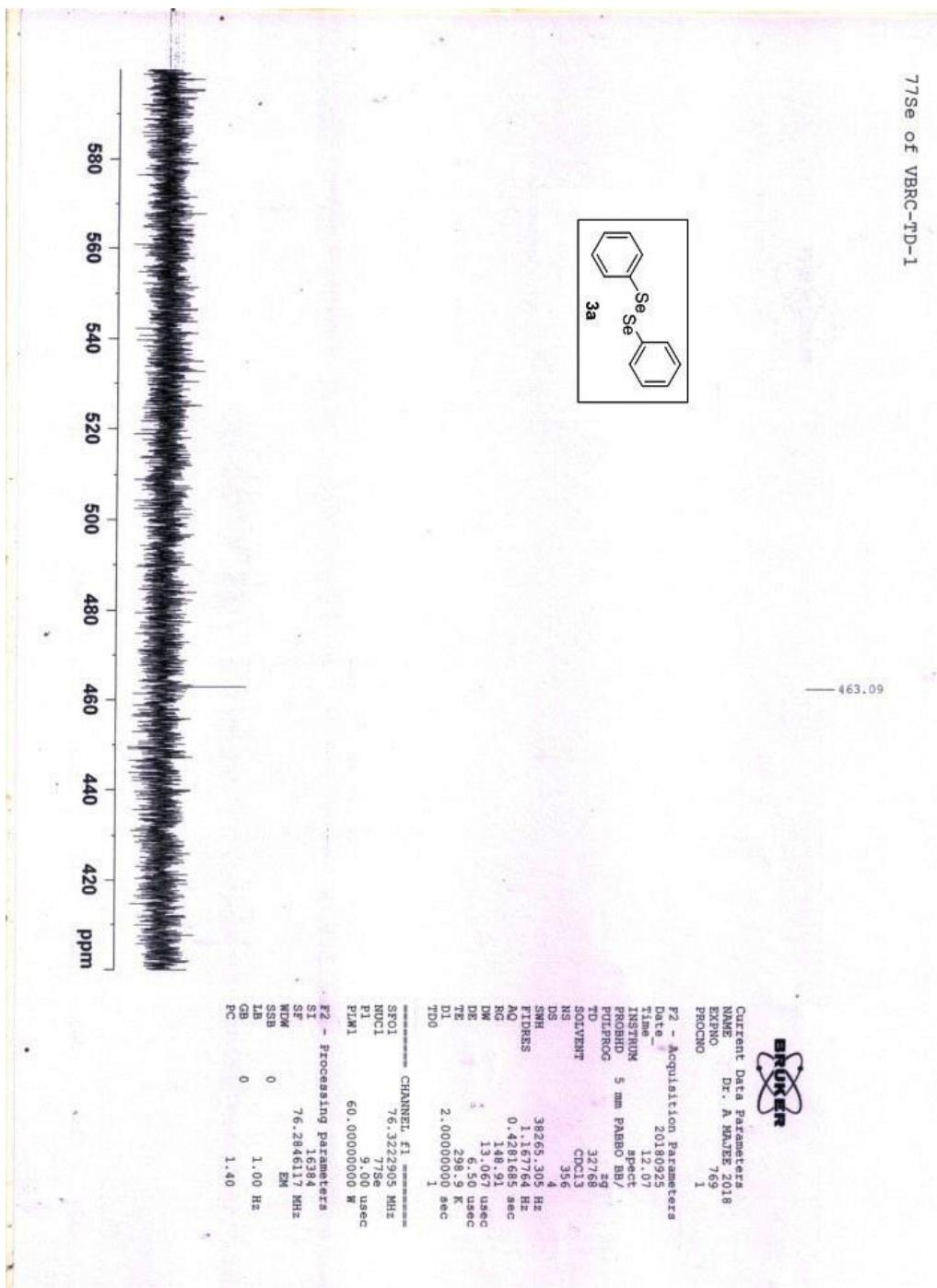


Fig. s1 ¹H NMR of **3a** in CDCl_3

**Fig. s2** ¹³C NMR of **3a** in CDCl₃

**Fig. s3** ⁷⁷Se NMR of **3a** in CDCl₃

1,2-di-p-tolyldiselenane (3b)¹: (261 mg, Yield: 77%); ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.52-7.50 (m, 4H), 7.10-7.04 (m, 4H), 2.37 (s, 6H); ¹³C NMR (CDCl₃, 100 MHz): δ (ppm) 138.1, 134.2, 132.4, 127.4, 21.2.

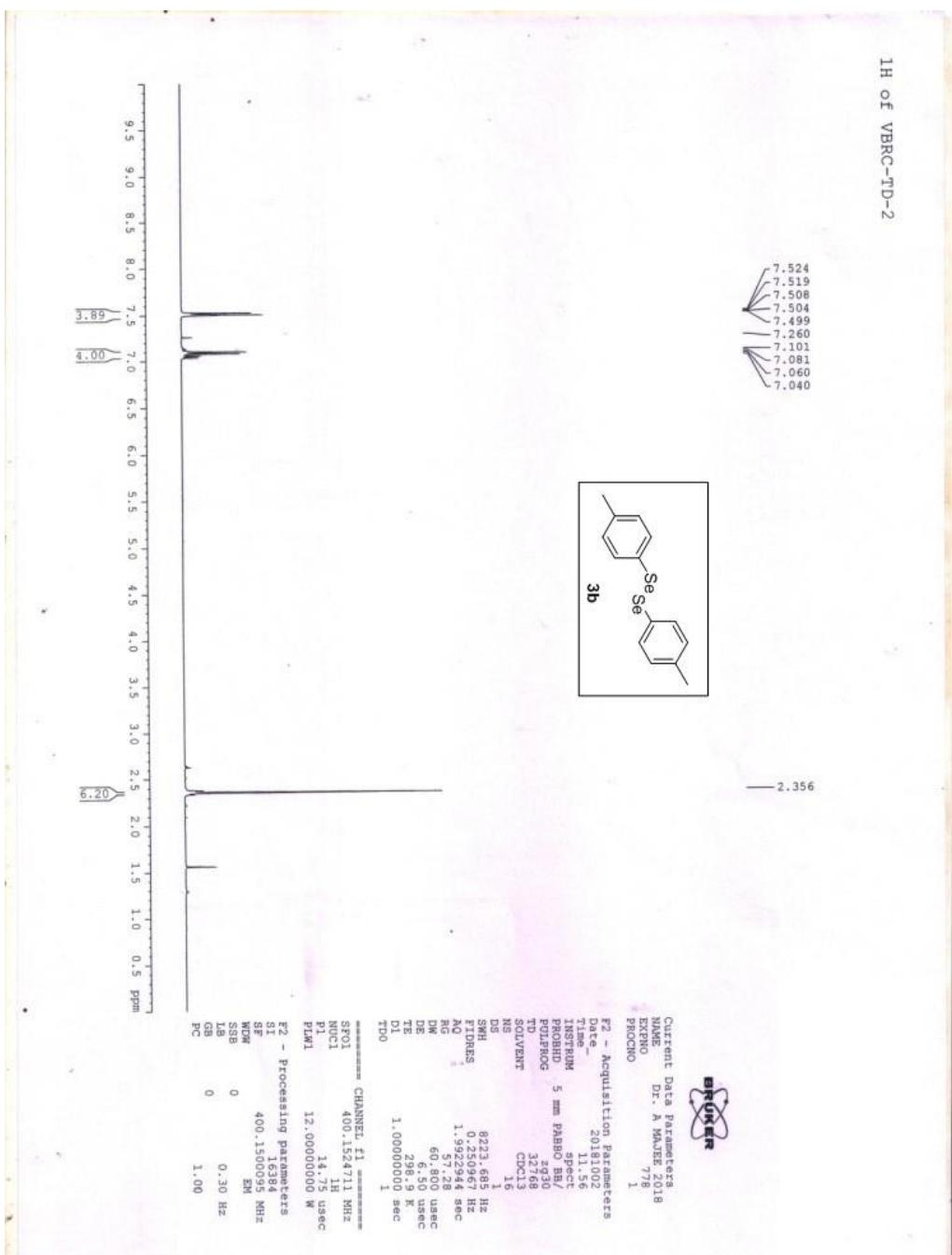
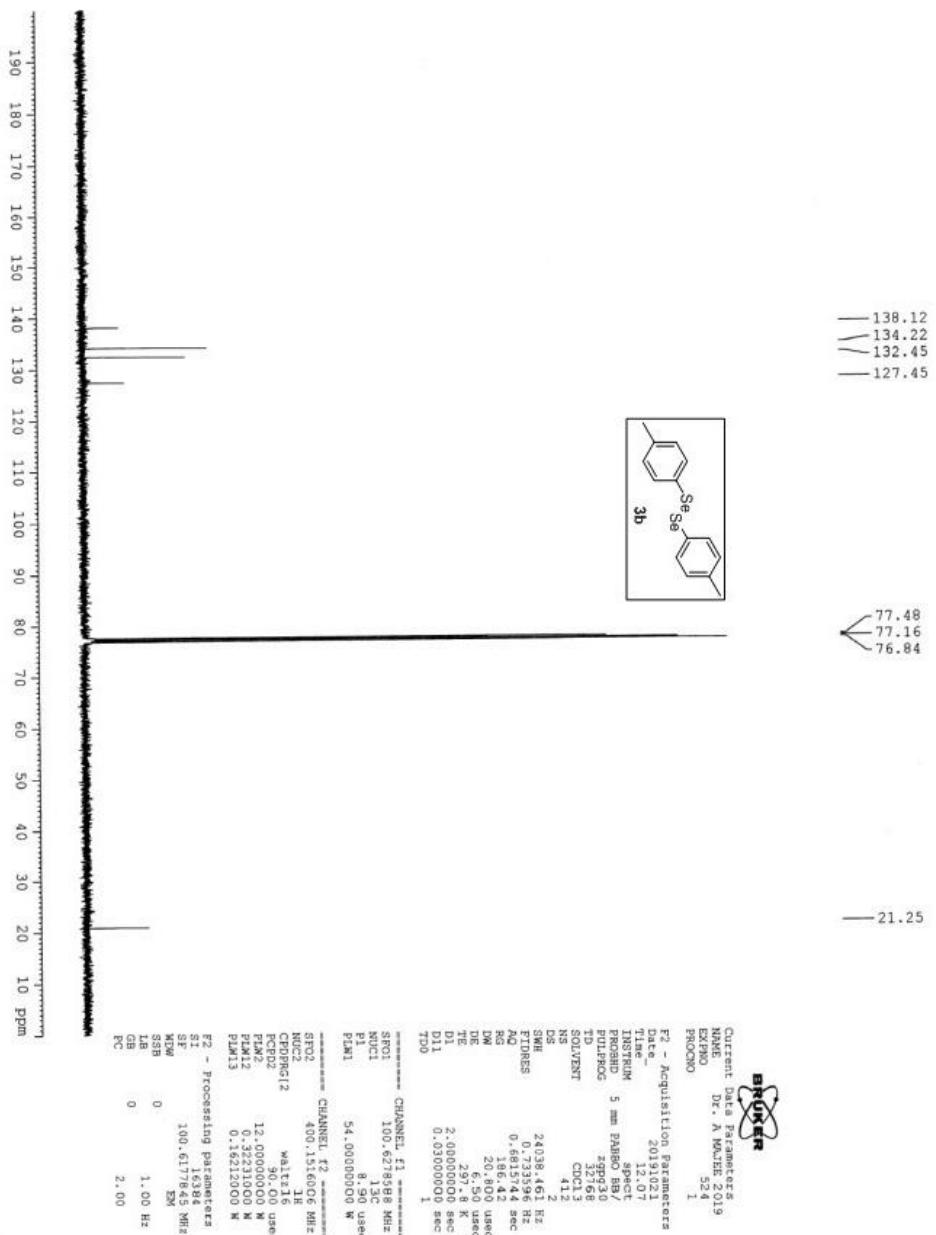


Fig. s4 ¹H NMR of **3b** in CDCl₃

**Fig. s5** ^{13}C NMR of **3b** in CDCl_3

1,2-di-m-tolyldiselenane (3c)¹: (255 mg, Yield: 75%); ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.43-7.41 (m, 4H), 7.18-7.14 (m, 2H), 7.07-7.05 (m, 2H), 2.32 (s, 6H); ¹³C NMR (CDCl₃, 100 MHz): δ (ppm) 139.1, 133.6, 132.3, 129.1, 128.8, 128.7, 21.4.

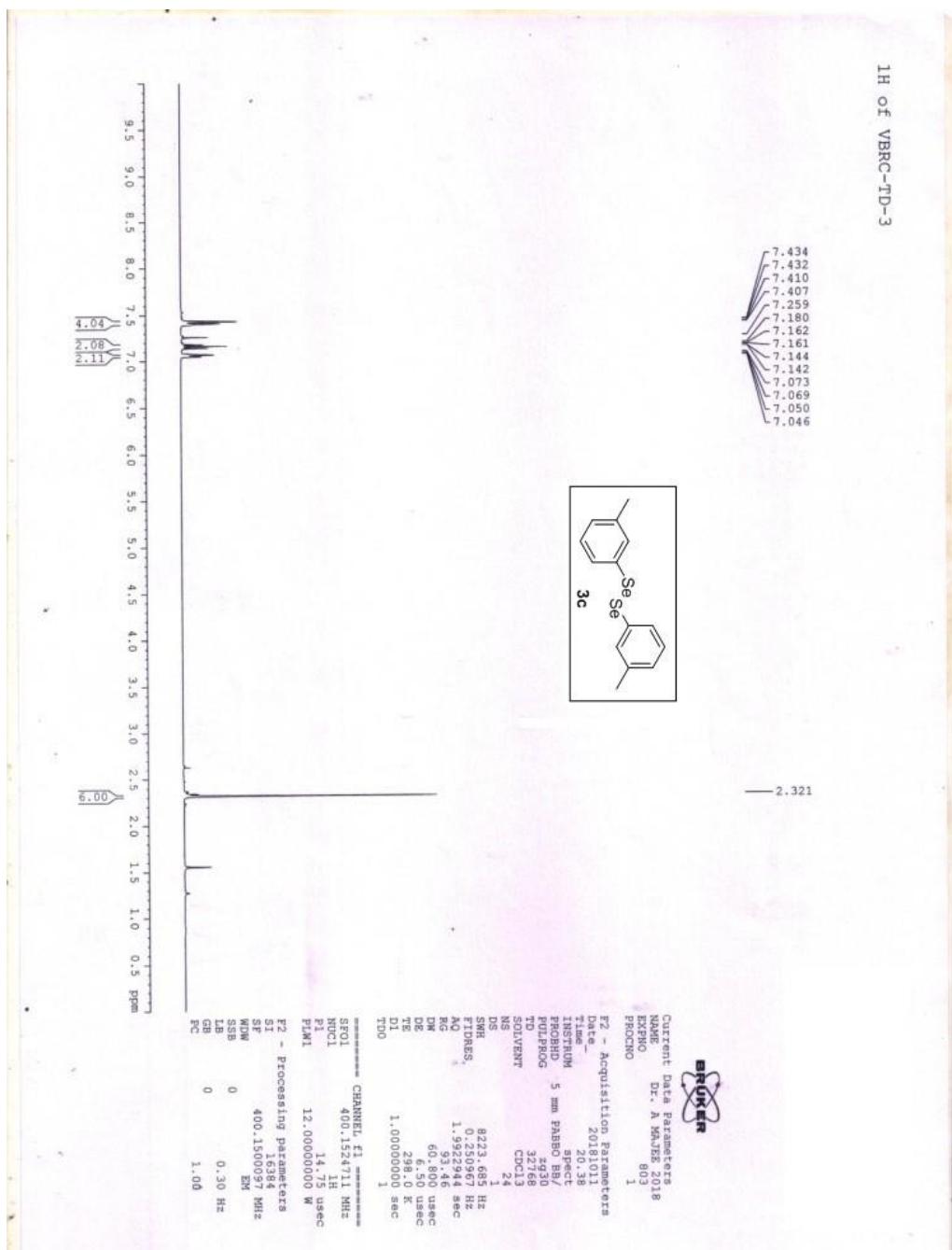
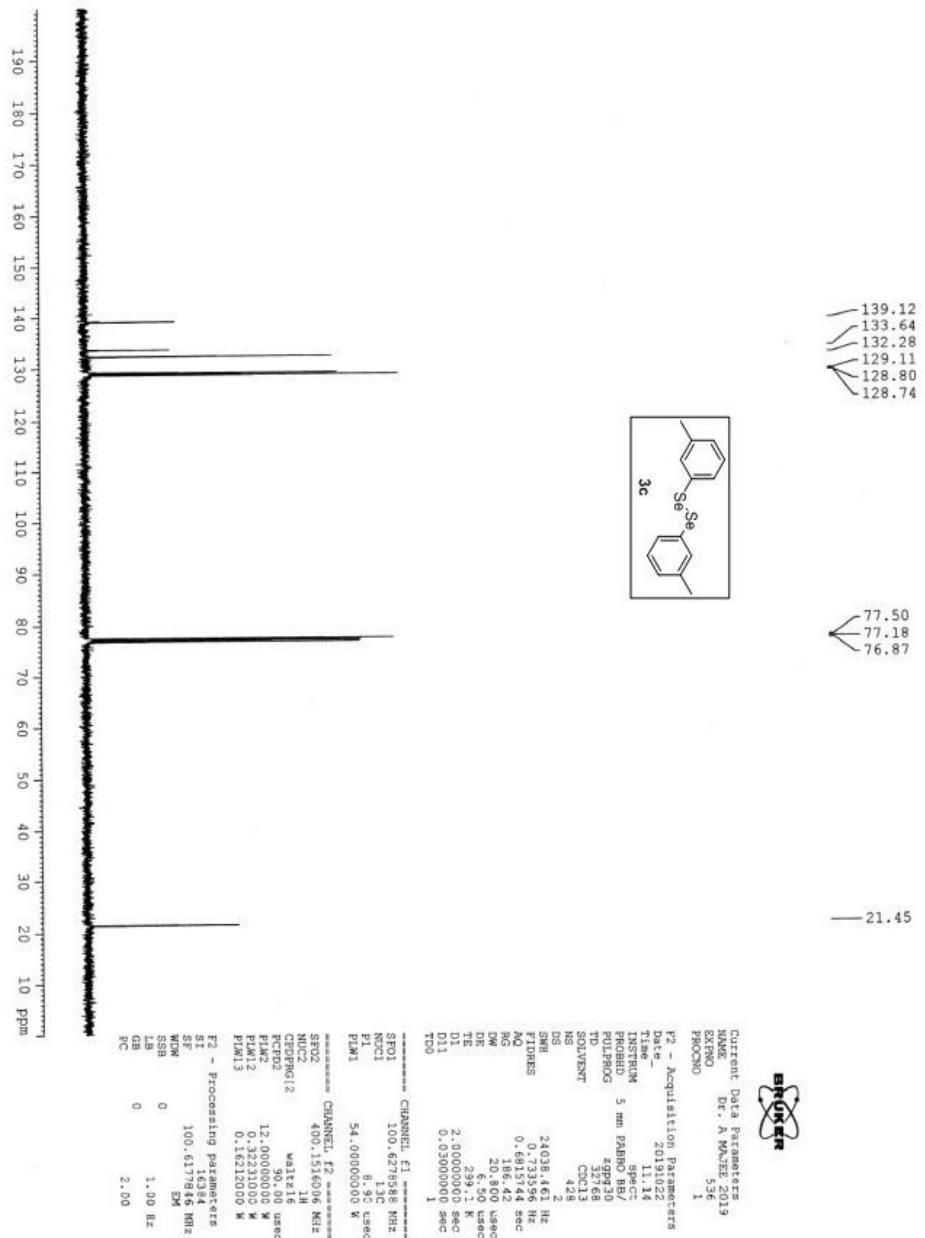


Fig. s6 ¹H NMR of **3c** in CDCl₃

Fig. s7 ¹³C NMR of **3c** in CDCl₃

1,2-bis(4-fluorophenyl)diselane (3d**)²:** (248 mg, Yield: 74%); ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.58-7.52 (m, 4H), 7.02-6.95 (m, 4H); ¹³C NMR (CDCl₃, 100 MHz): δ (ppm) 163.2 (d, *J*_{C-F} = 244 Hz), 135.0 (d, *J*_{C-F} = 7 Hz), 116.6 (d, *J*_{C-F} = 4 Hz), 116.4 (d, *J*_{C-F} = 3 Hz).

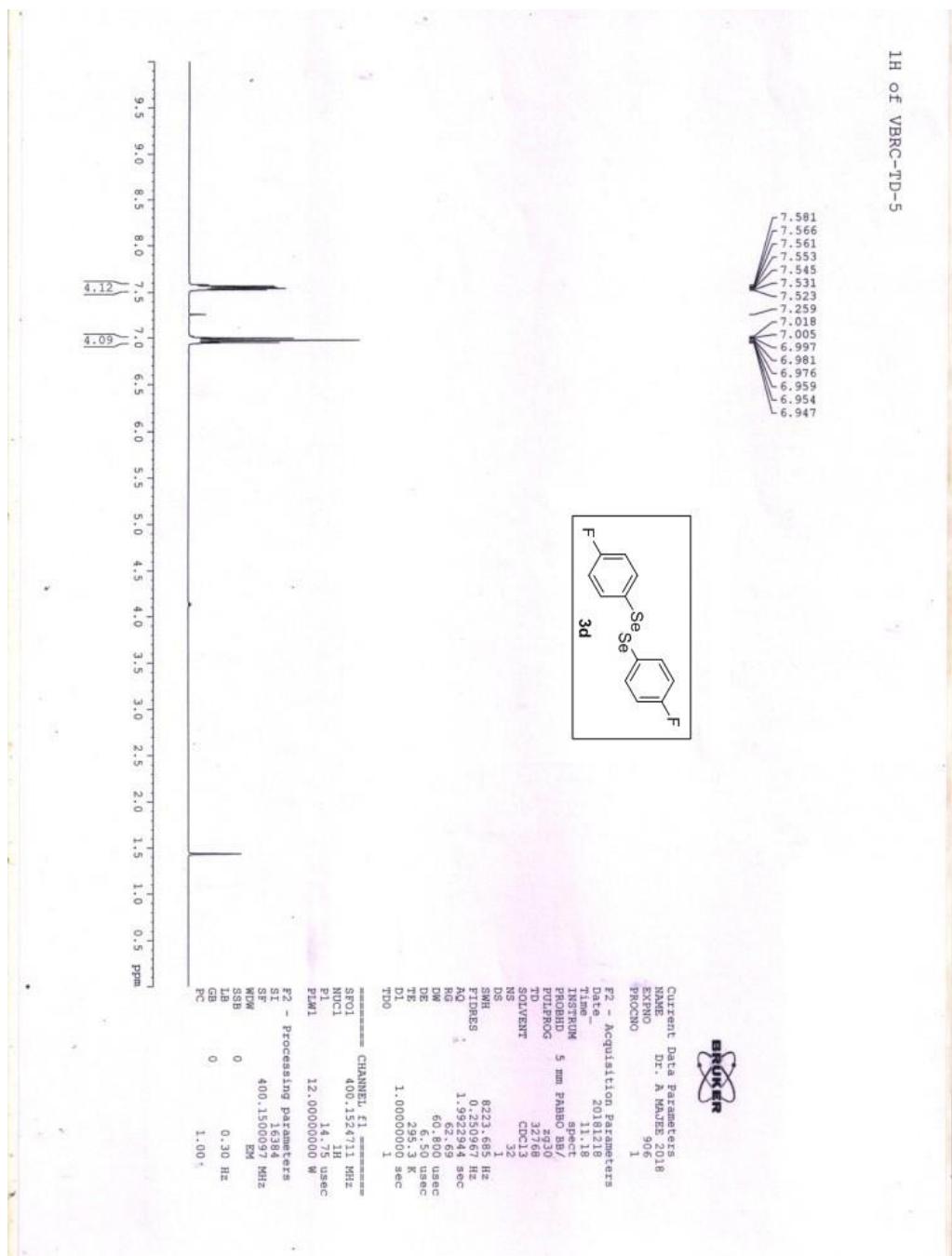
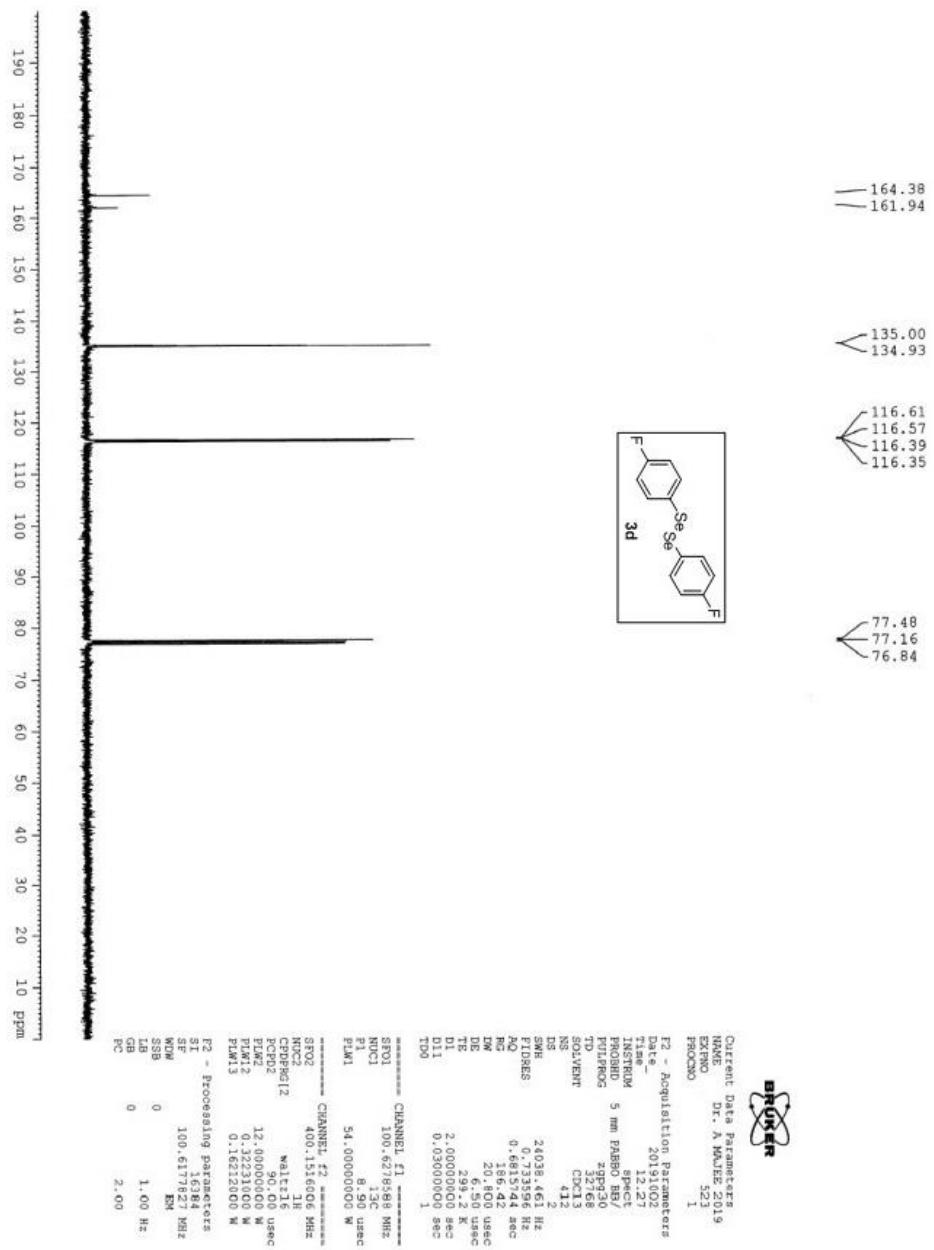


Fig. s8 ¹H NMR of **3d** in CDCl₃

**Fig. s9** ¹³C NMR of **3d** in CDCl₃

1,2-bis(4-chlorophenyl)diselane (3e)¹: (289 mg, Yield: 76%); ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.52-7.49 (m, 4H), 7.25-7.22 (m, 4H); ¹³C NMR (CDCl₃, 100 MHz): δ (ppm) 134.4, 133.4, 129.7, 129.5.

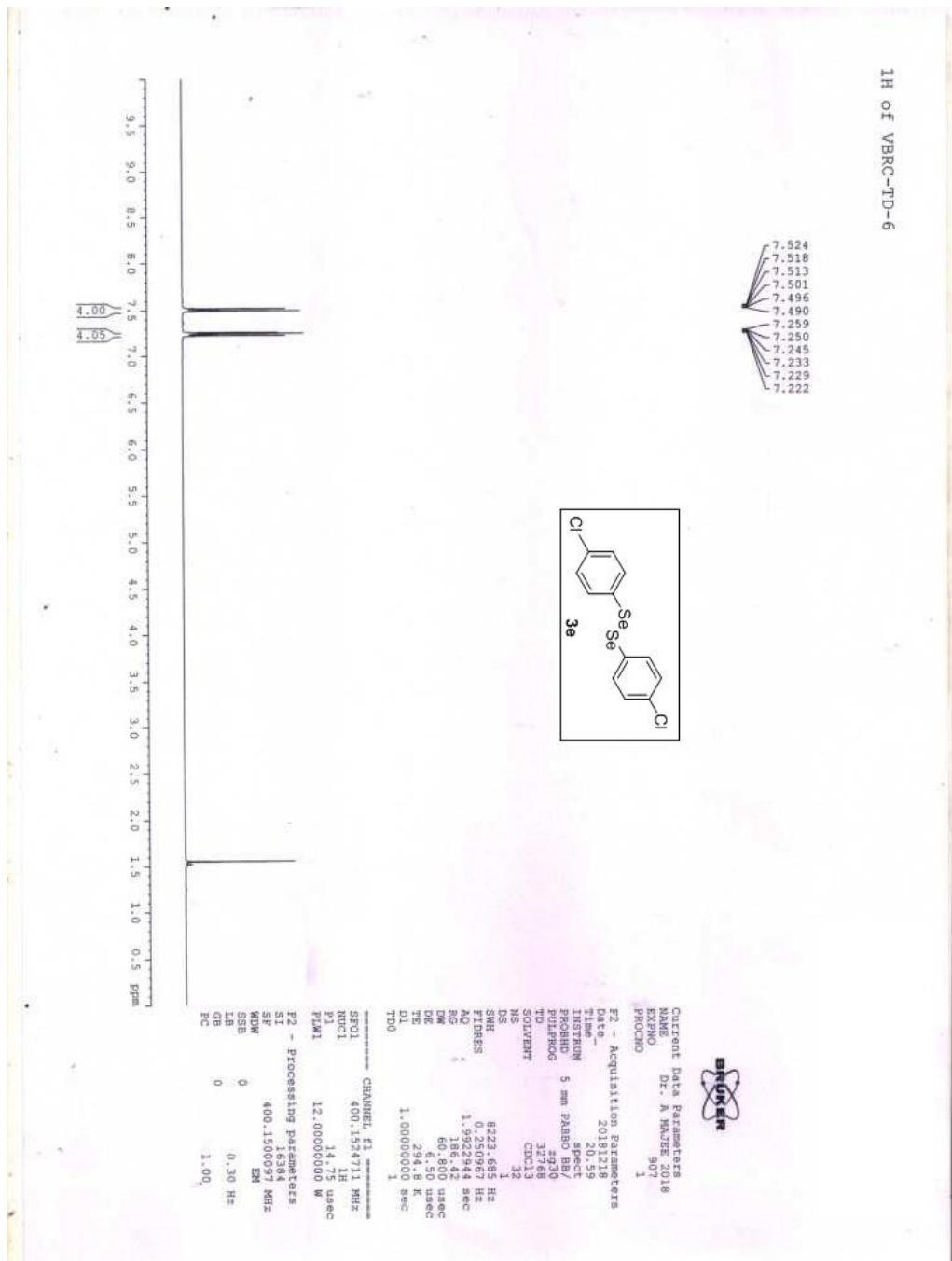


Fig. s10 ¹H NMR of 3e in CDCl₃

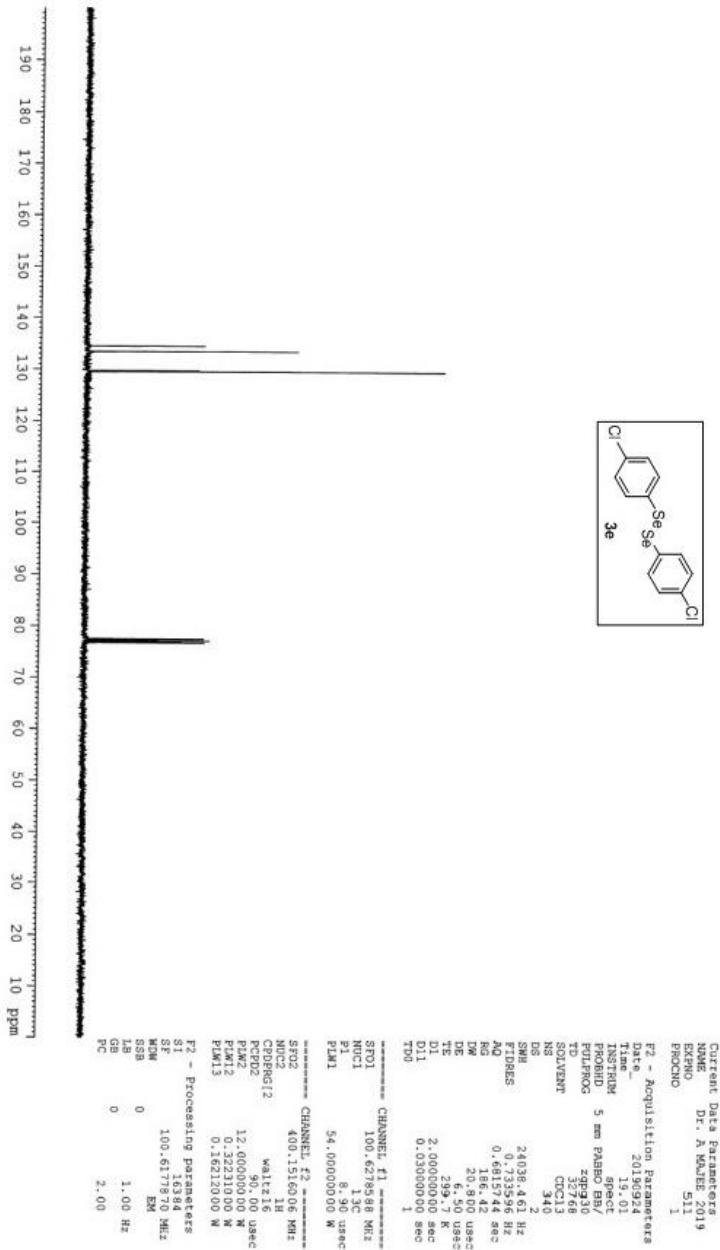


Fig. s11 ¹³C NMR of 3e in CDCl₃

1,2-bis(4-bromophenyl)diselane (3f)¹: (353 mg, Yield: 75%); ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.45-7.37 (m, 8H); ¹³C NMR (CDCl₃, 100 MHz): δ (ppm) 134.7, 133.5, 132.4, 122.4.

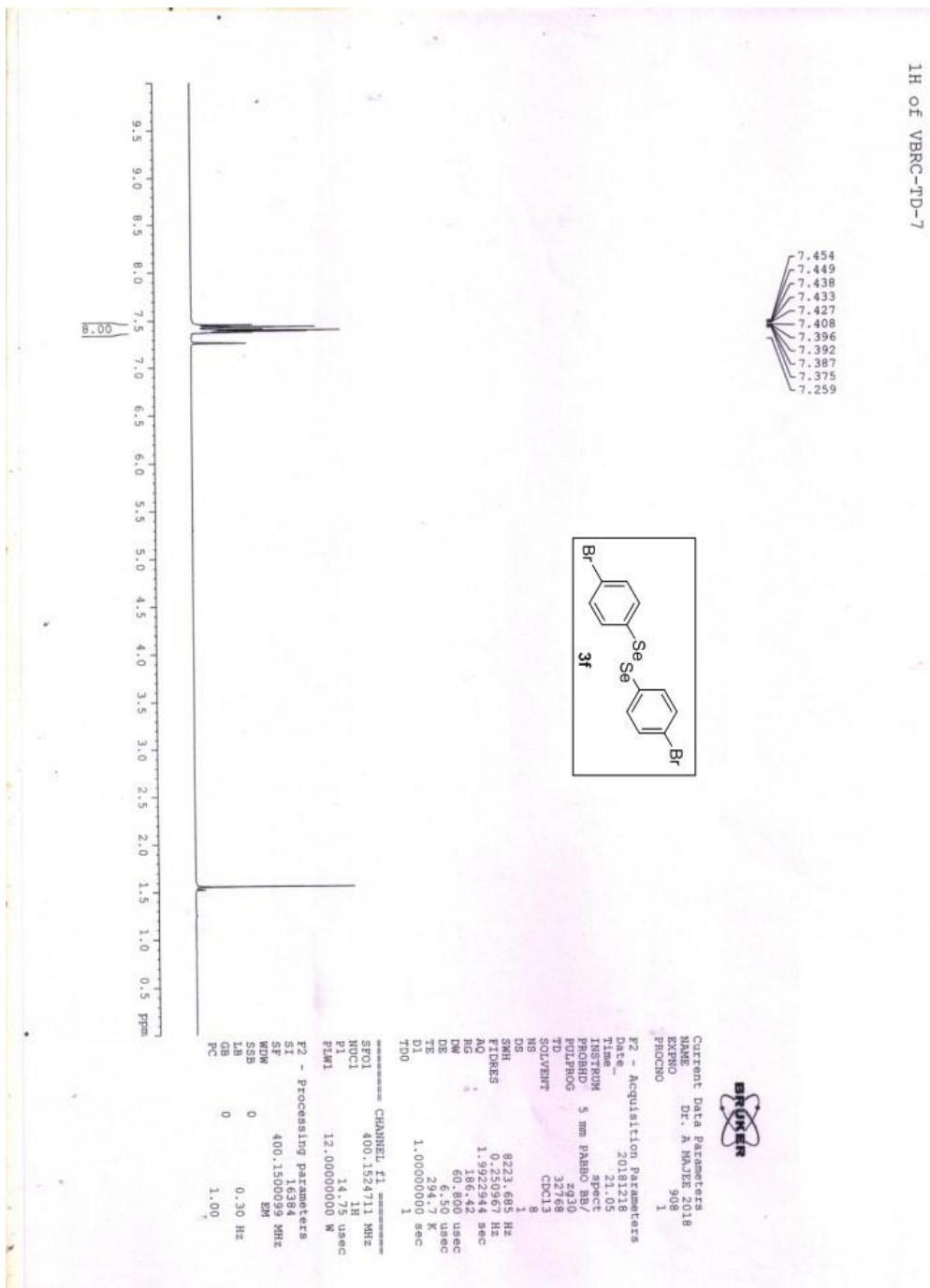
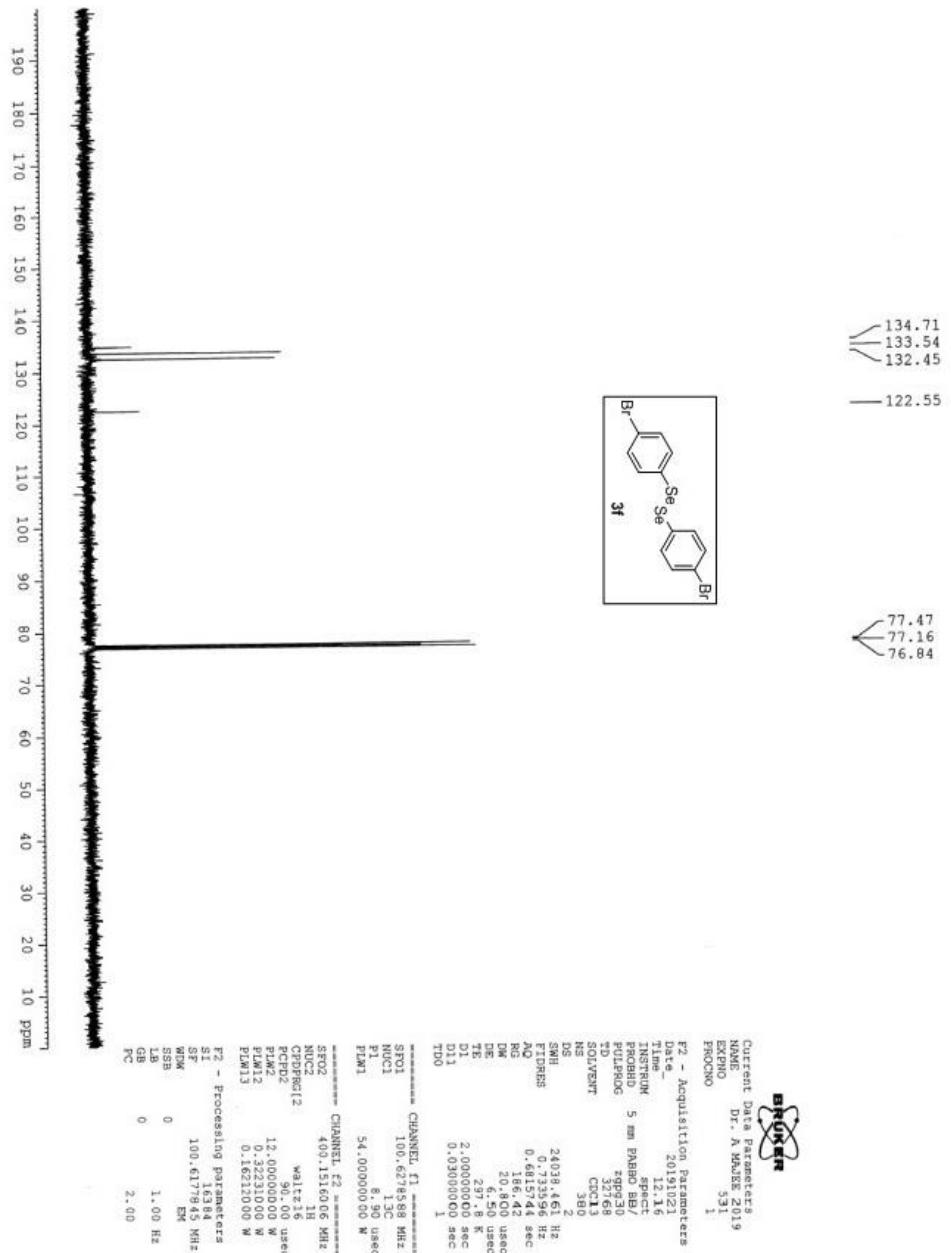


Fig. s12 ¹H NMR of 3f in CDCl₃

Fig. s13 ¹³C NMR of **3f** in CDCl₃

1,2-bis(3-(trifluoromethyl)phenyl)diselane (3g)³: (323mg, Yield: 72%); ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.92-7.88 (m, 1H), 7.82-7.77 (m, 2H), 7.62-7.55 (m, 3H), 7.47-7.41 (m, 2H), ¹³C NMR (CDCl₃, 100 MHz): δ (ppm) 136.4, 135.0, 131.5 (2C), 130.0, 129.8, 129.6, 128.5 (q, J_{C-F} = 11 Hz), 125.0 (2C), 124.8 (2C), 122.3, 119.6.

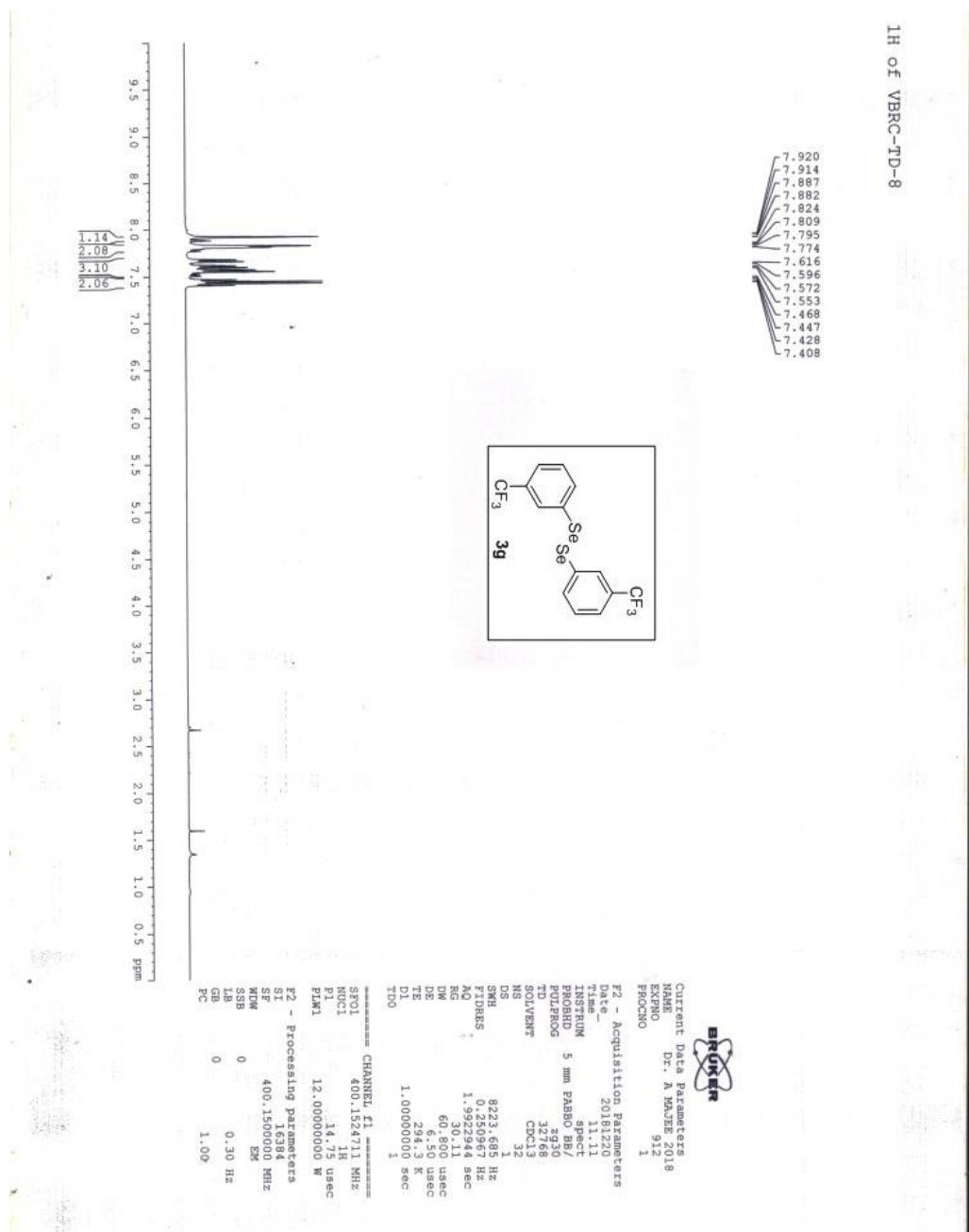
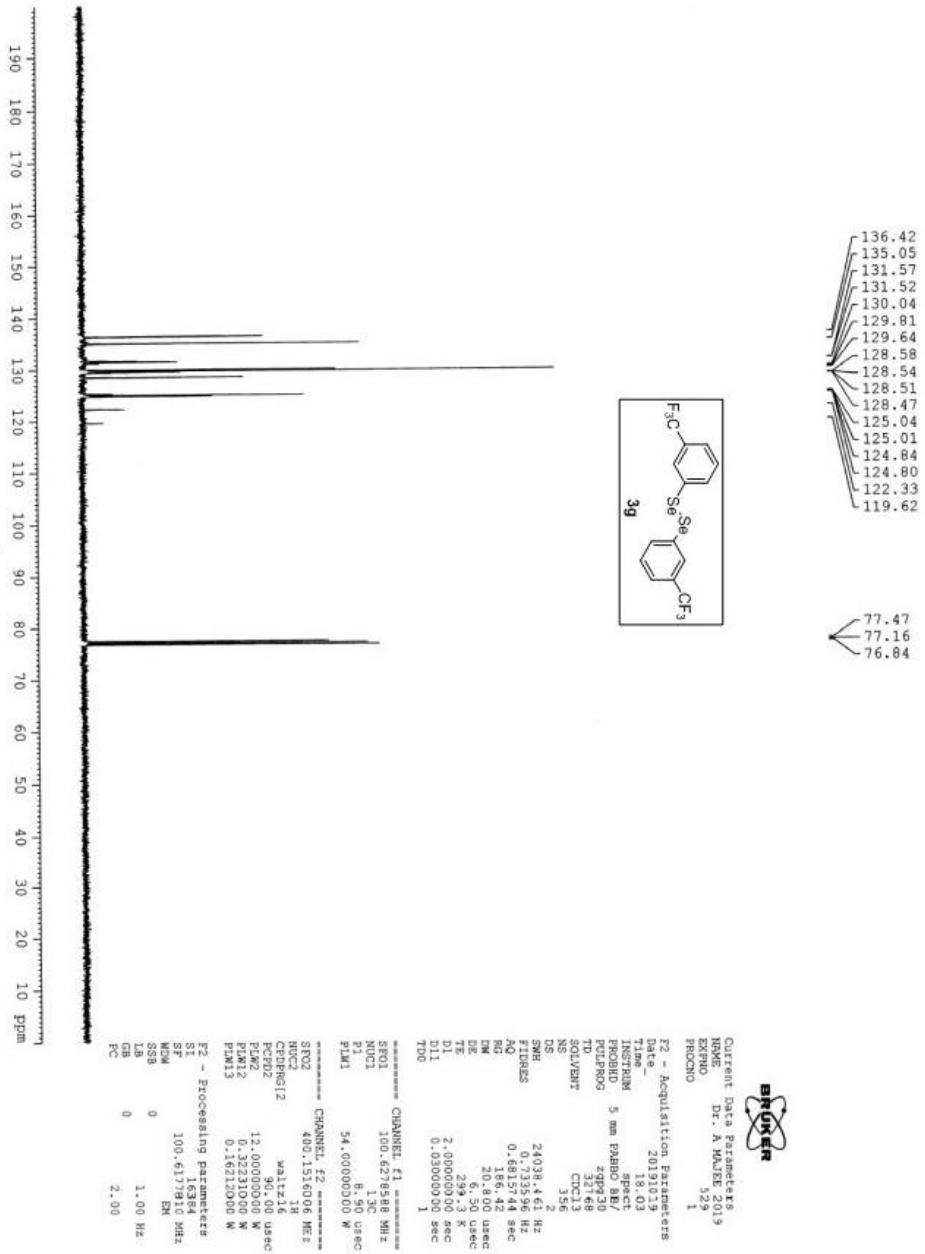


Fig. s14 ¹H NMR of 3g in CDCl₃

Fig. s15 ¹³C NMR of **3g** in CDCl₃

1,2-di(thiophen-2-yl)diselane (3h**)⁴:** (237 mg, Yield: 73%); ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 7.50-7.48 (m, 2H), 7.24-7.23 (m, 2H), 7.02-7.00 (m, 2H); ¹³C NMR (CDCl₃, 100 MHz): δ (ppm) 137.1, 134.8, 131.1, 128.0.

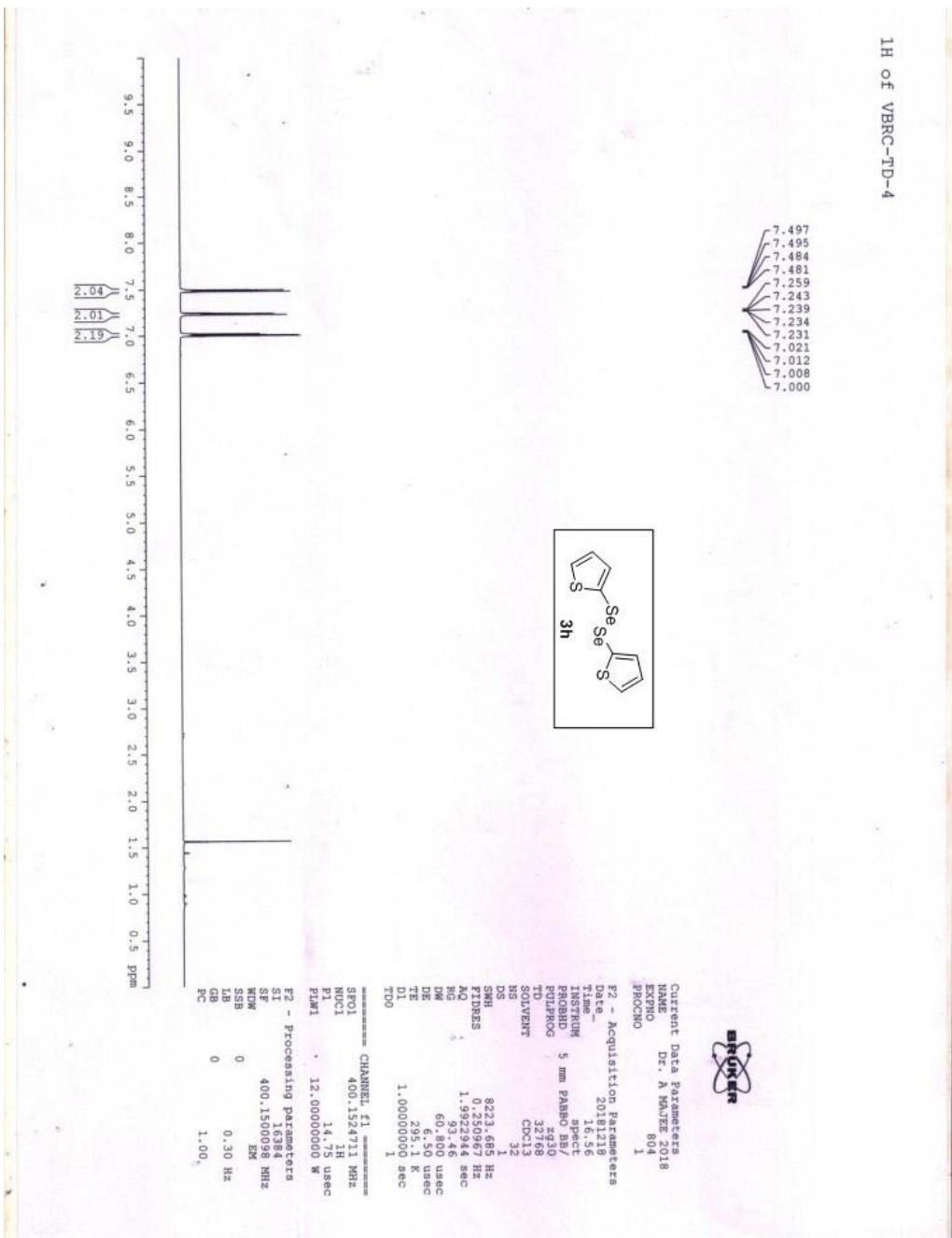


Fig. s16 ¹H NMR of **3h** in CDCl₃

13C of VBRC-2

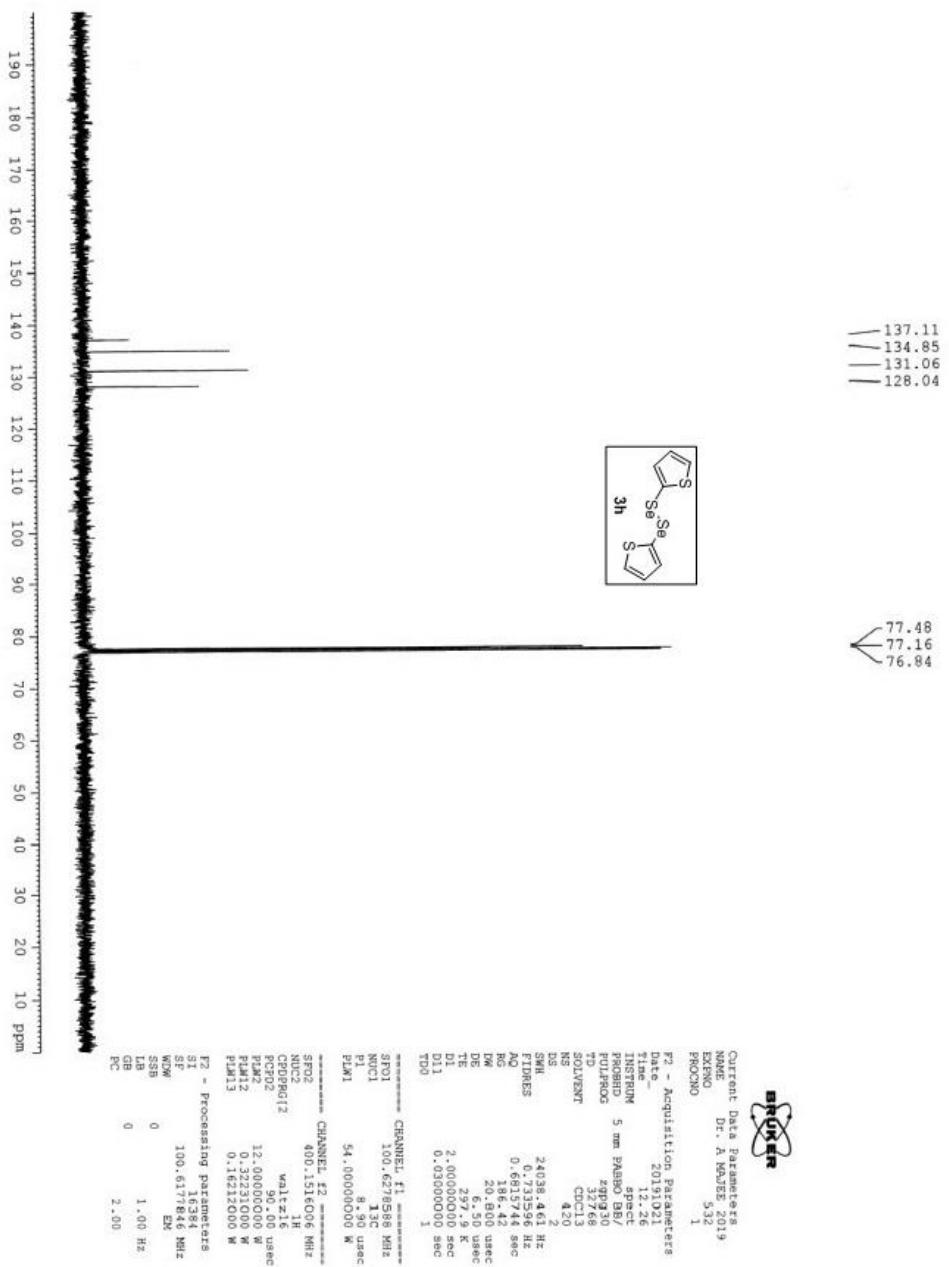


Fig. s17 ¹³C NMR of **3h** in CDCl₃

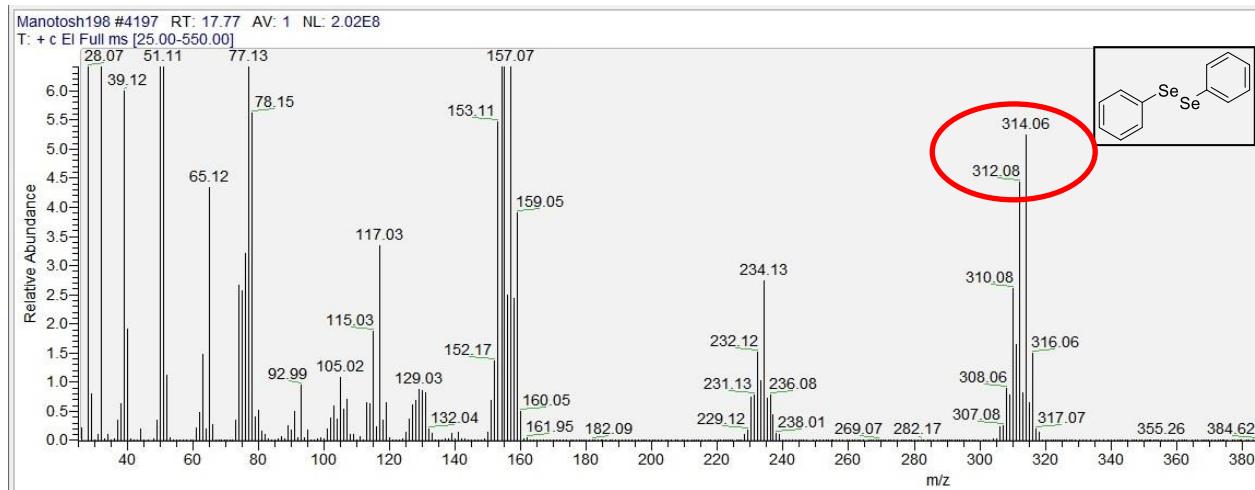


Fig. s18 Mass spectra of **3a**

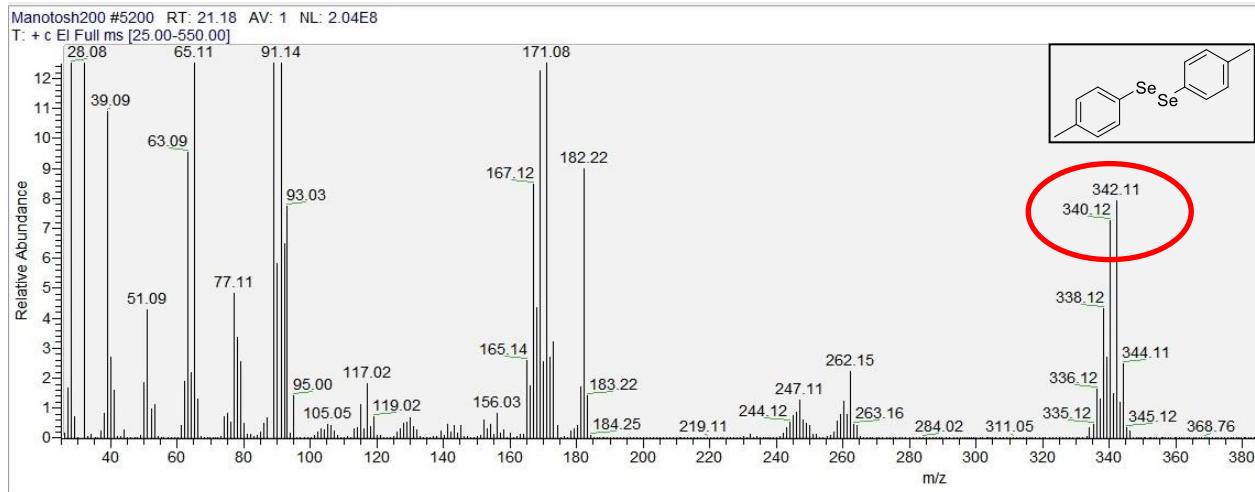


Fig. s19 Mass spectra of **3b**

References

- | | |
|----|--|
| 1. | Singh, D.; Deobald, A. M.; Camargo, L. R. S.; Tabarelli, G.; Rodrigues, O. E. D.; Braga, A. L. An Efficient One-Pot Synthesis of Symmetrical Diselenides or Ditellurides from Halides with CuO Nanopowder/Se ⁰ or Te ⁰ /Base. <i>Org. Lett.</i> 2010 , <i>12</i> , 3288-3291. |
| 2. | Li, Z.; Ke, F.; Deng, H.; Xu, H.; Xiang, H.; Zhou, X. Synthesis of disulfides and diselenides by copper-catalyzed coupling reactions in water. <i>Org. Biomol. Chem.</i> 2013 , <i>11</i> , 2943-2946. |
| 3. | Curran, S.P.; Connon, S.J. Selenide Ions as Catalysts for Homo- and Crossed-Tishchenko Reactions of Expanded Scope. <i>Org. Lett.</i> 2012 , <i>14</i> , 1074-1077. |
| 4. | Balkrishna, S.J.; Bhakuni, B.S.; Kumar, S. Copper catalyzed/mediated synthetic methodology for ebselen and related isoselenazolones. <i>Tetrahedron</i> 2011 , <i>67</i> , 9565-9575. |