Supporting Information

Application of One-Pot Three-Component Condensation Reaction for the Synthesis of New Organo Phosphorus-Sulfur Macrocycles

*Guoxiong Hua, David B. Cordes, Alexandra M. Z. Slawin and J. Derek Woollins**

1. Experimental Section

Unless otherwise stated, all reactions were carried out under on oxygen free nitrogen atmosphere using pre-dried solvents and standard Schlenk techniques, subsequent chromatographic and work up procedures were performed in air. All chemicals and Lawesson's reagent were purchased from Sigma-Aldrich without further purification. 2,4-diferrocenyl-1,3,2,4-diathiadiphosphetane 2,4-disulfide $[FcP(\mu\text{-}S)S]_2$ (**FcLR)**, was prepared by using the literature method.[s1,s2] ^1H (400.1 MHz), ^{13}C (100.6 MHz) and $^{31}P\text{-}\{^1H\}$ (162.0 MHz) NMR spectra were recorded at 25 $^{\circ}C$ (unless stated otherwise) on Bruker Advance II 400. IR spectra were recorded as KBr pellets in the range of 4000-250 cm^{-1} on a Perkin-Elmer 2000 FTIR/Raman spectrometer. Mass spectrometry was performed by the EPSRC National Mass Spectrometry Service Centre, Swansea. X-ray diffraction data for compound **14** was collected at 125 K using the St Andrews Automated Robotic Diffractometer (STANDARD),[s3] consisting of a sealed-tube generator (Rigaku, Houston, USA) equipped with a SHINE monochromator [Mo Kα radiation (λ = 0.71075 Å)], and a Saturn 724 CCD area detector, coupled with a Microglide goniometer head and an ACTOR SM robotic sample changer. Diffraction data for compounds **10a**, **10b**, **11**, **12a** and **12b** were collected at either 93 K or 173 K (all other crystals) by using a Rigaku FR-X Ultrahigh Brilliance Microfocus RA generator/confocal optics and Rigaku XtaLAB P200 system, with Mo Kα radiation (λ = 0.71075 Å). Intensity data were collected using ω steps accumulating area detector images spanning at least a hemisphere of reciprocal space. All data were corrected for Lorentz polarization effects. A multi-scan absorption correction was applied by using CrystalClear.[s4,s5] Structures were solved by either charge-flipping (SUPERFLIP)[s6] or direct (SIR2004, SIR2011)[s7,s8] methods, and refined by full-matrix least-squares against F^2 (SHELXL-2013).[s9] Non-hydrogen atoms were refined anisotropically, and hydrogen atoms were refined using a riding model. All calculations were performed using the CrystalStructure interface.[s10]

2. Synthesis and Characterization of Heterocycles 1-12 and 14

General Procedure for Preparation of Macrocycles 1-12 and 14. A suspension of alkenyl- or aryl-dithiol (1.0 mmol), triethylamine (2.0 mmol), **LR** (0.44g, 1.0 mmol) or **FcLR** (0.56 g, 1.0 mmol) and dihaloalkane (1.0 mmol) in dry THF (60 mL) was stirred under N_2 gas atmosphere at ambient temperature for 24 h leading to a reddish yellow or pale-yellow suspension. Upon filtering to remove insoluble solid the filtrate was dried under reduced pressure. The residue was dissolved in dichloromethane (*ca*. 2 mL) and was loaded onto a silica gel column (dichloromethane as eluent) to give the macrocycles. Another unexpected nine-membered ring **4** was also obtained in respective 25% and 30% yields in the cases of **5** and **9**. Two diastereomers were separated completely in the cases of **10** and **12**.

Compound 1. White solid (80% yield). Two diastereoisomers were found in *ca.* 1:1 intensity ratio. Selected IR (KBr, cm^{-1}): 1590(s), 1564(m), 1495(s), 1407(m), 1296(m), 1260(s), 1179(m), 1098(s), 1025(m), 829(m), 701(s), 618(m), 528(s). ^{1}H NMR (CD_2Cl_2, δ), 8.03-7.71 (m, 8H), 8.03-7.71 (m, 8H), 7.29-6.88 (m, 8H), 4.09-3.96 (m, 8H), 3.78 (s, 6H), 3.76 (s, 6H) ppm. ^{13}C NMR (CD_2Cl_2, δ), 165.4, 164.2, 134.9, 133.7, 133.6, 133.4, 131.8, 131.7, 129.1, 128.8, 115.0, 114.9, 114.8, 114.7, 56.1, 56.0, 35.9, 35.7 ppm. ^{31}P NMR (CD_2Cl_2, δ), 95.1 and 95.0 ppm. Mass spectrum (EI^+, m/z), 656 $[M]^+$. Accurate mass measurement [EI^+, m/z]: 655.9196 $[M]^+$, calculated mass for $C_{24}H_{22}N_2O_2P_2S_7$: 655.9201.

Compound 2. White solid (60% yield). Two diastereomers were found in *ca.* 1:1 intensity ratio. Selected IR (KBr, cm^{-1}): 1590(vs), 1564(m), 1495(s), 440(m), 1407(m), 1295(m), 1259(vs), 1179(s), 1097(vs), 1024(s), 828(m), 800(m), 699(s), 618(m), 527(s), 444(m). ^{1}H NMR (CD_2Cl_2, δ), 7.97-7.84 (m, 2H), 7.38-6.98 (m, 22H), 4.51-3.86 (m, 8H), 3.94 (s, 6H), 3.93 (s, 6H) ppm. ^{13}C NMR (CD_2Cl_2, δ), 164.4, 164.2, 138.4, 138.3, 133.9, 133.7, 133.5, 133.3, 131.9, 131.8, 131.2, 130.8, 130.0, 129.9, 129.4, 129.1, 129.0, 128.8, 56.1, 55.9, 38.9, 38.1 ppm. ^{31}P NMR (CD_2Cl_2, δ), 83.7 and 82.7 ppm. Mass spectrum (EI^+, m/z), 656 $[M]^+$. Accurate mass measurement [EI^+, m/z]: 655.9198 $[M]^+$, calculated mass for $C_{24}H_{22}N_2O_2P_2S_7$: 655.9201.

Compound 3. White solid (58% yield). Two diastereomers were found in *ca.* 1:2 intensity ratio. Selected IR (KBr, cm^{-1}): 1591(s), 1564(m), 1495(s), 1459(m), 1406(m), 1293(m), 1256(s), 1178(s), 1098(s), 1023(s), 828(m), 799(m), 687(s), 618(m), 533(s), 501(m). ^{1}H NMR (CD_2Cl_2, δ), 7.89-7.86 (m, 8H), 7.47-6.93 (m, 16H), 4.44-4.33 (m, 4H), 4.22-4.02 (m, 4H), 3.84 (s, 6H), 3.82 (s, 6H), 3.72-3.65 (m, 4H), 3.18-2.29 (m, 4H) ppm. ^{13}C NMR (CD_2Cl_2, δ), 163.2, 137.2, 135.9, 133.0, 132.8, 131.9, 131.7, 130.7, 130.2, 129.9, 129.5, 129.3, 128.3, 114.3, 114.0, 55.7, 42.8, 37.7, 34.1, 25.3 ppm. ^{31}P NMR (CD_2Cl_2, δ), 79.9 and 79.7 ppm. Mass spectrum (EI^+, m/z), 600 $[M]^+$. Accurate mass measurement [EI^+, m/z]: 599.9729 $[M]^+$, calculated mass for $C_{24}H_{26}O_2P_2S_6$: 599.9727.

Compound 5. Yellow solid (53% yield). Two diastereomers were found in *ca.* 1:1 intensity ratio. Selected IR (KBr, cm^{-1}): 1484(m), 1408(m), 1258(m), 1168(s), 1105(m), 1022(s), 1000(m), 822(s), 766(m), 734(m), 672(vs), 616(m), 531(m), 474(vs). ^{1}H NMR (CD_2Cl_2, δ), 7.42-7.29 (m, 8H), 4.77-4.74 (m, 8H), 4.67-4.60 (m, 8H), 4.43 (s, 10H), 4.40 (s, 10H), 4.33-4.27 (m, 4H), 4.11-4.03 (m, 4H), 3.71-3.52 (m, 4H), 3.31-2.90 (m, 4H) ppm. ^{13}C NMR (CD_2Cl_2, δ), 134.8 (d, J(P,C) = 11.4 Hz), 134.5 (d, J(P,C) = 10.8 Hz), 132.4 (d, J(P,C) = 6.6 Hz), 131.0, 129.5 (d, J(P,C) = 7.4 Hz), 129.1, 79.3 (d, J(P,C) = 101 Hz), 79.0 (d, J(P,C) = 101 Hz), 72.1 (d, J(P,C)= 11.9 Hz), 72.0 (d, J(P,C)= 12.1 Hz), 72.4 (d, J(P,C)= 15.7 Hz), 72.0 (d, J(P,C)= 14.9 Hz), 71.7, 71.5, 37.1, 35.4, 34.6, 34.5 ppm. ^{31}P NMR (CD_2Cl_2, δ), 79.6 and 79.1 ppm. Mass spectrum (EI^+, m/z), 756 $[M]^+$. Accurate mass measurement [EI^+, m/z]: 755.8835 $[M]^+$, calculated mass for $C_{30}H_{30}Fe_2P_2S_6$: 755.8840.

Compound 6. Orange solid (46% yield). Two diastereomers were found in *ca.* 1:2 intensity ratio. Selected IR (KBr, cm^{-1}): 1484(m), 1408(m), 1258(m), 1170(s), 1105(m), 1024(s), 1000(m), 951(m), 824(s), 706(s), 676(vs), 530(m), 474(s). ^{1}H NMR (CD_2Cl_2, δ), 7.66 (s, 2H), 7.22-7.16 (m, 6H), 4.58-4.55 (m, 8H), 4.47-4.43 (m, 8H), 4.30 (s, 10H), 4.29 (s, 10H), 4.18-3.96 (m, 4H), 3.66-3.61 (m, 4H), 3.15-2.66 (m, 8H) ppm. ^{13}C NMR (CD_2Cl_2, δ), 139.2, 138.9, 130.5, 130.3, 128.4, 128.3, 127.2, 126.7, 80.2 (d, J(P,C) = 103 Hz), 79.9 (d, J(P,C) = 103 Hz), 72.1 (d, J(P,C) = 12.1 Hz), 72.0 (d, J(P,C) = 11.7 Hz), 71.4 (d, J(P,C) = 15.3 Hz), 71.3 (d, J(P,C) = 14.9 Hz), 71.0, 70.8, 38.3, 37.7, 32.7, 32.6 ppm. ^{31}P NMR (CD_2Cl_2, δ), 82.3 and 82.2 ppm. Mass spectrum (EI^+, m/z), 756 $[M]^+$. Accurate mass measurement [EI^+, m/z]: 755.8850 $[M]^+$, calculated mass for $C_{30}H_{30}Fe_2P_2S_6$: 755.8846.

Compound 8. Dark yellow solid (40% yield). Two diastereomers were found in *ca.* 1:1 intensity ratio. Selected IR (KBr, cm^{-1}): 1310(m), 1232(m), 1172(s), 1106(m), 1024(s), 824(s), 705(vs), 673(vs), 530(m), 477(vs). ^{1}H NMR (CD_2Cl_2, δ), 7.34-7.10 (m, 16H), 4.62-4.60 (m, 16H), 4.32 (s, 10H), 4.31 (s, 10H), 4.11-3.89 (m, 8H), 3.66-3.55 (m, 8H) ppm. ^{13}C NMR (CD_2Cl_2, δ), 137.8, 137.7, 130.8, 130.2, 129.4, 128.9, 128.6, 128.3, 78.4 (d, J(P,C) = 101 Hz), 78.2 (d, J(P,C) = 102 Hz), 72.4, 72.3, 71.9, 71.7, 71.2, 70.9, 38.0, 35.6 ppm. ^{31}P NMR (CD_2Cl_2, δ), 82.0 and 81.4 ppm. Mass spectrum (CI^+, m/z), 833 $[M+H]^+$. Accurate mass measurement [CI^+, m/z]: 832.9233 $[M+H]^+$, calculated mass for $C_{36}H_{34}Fe_2P_2S_6H$: 832.9237.

Compound 9. Golden solid (65% yield). Four diastereomers were found in *ca.* 1:1:2:2 intensity ratio. Selected IR (KBr, cm^{-1}): 1583(m), 1454(m), 1409(m), 1384(m), 1169(s), 1106(m), 1023(s), 1002(m), 821(s), 766(m), 674(vs), 535(m), 478(vs). ^{1}H NMR (CD_2Cl_2, δ), 8.00 (s, 2H), 7.98 (s, 1H), 7.96 (s, 1H), 7.47-7.10 (m, 24H), 4.72-4.63 (m, 8H), 4.51-4.45 (m, 8H), 4.36 (s, 5H), 4.32 (s, 5H), 4.30 (s, 5H), 4.25 (s, 5H), 4.14-3.84 (m, 16H), 2.28 (s, 3H), 2.27 (s, 3H), 2.24 (s, 3H), 2.23 (s, 3H) ppm. ^{13}C NMR (CD_2Cl_2, δ), 141.2, 140.2, 139.0, 138.5, 137.9, 137.6, 136.0, 135.9, 135.7, 135.5, 135.2, 135.1, 135.0, 131.7, 131.4, 131.3, 130.6, 130.2,

129.0, 128.7, 128.5, 126.9, 126.8, 126.1, 126.0, 82.0 (d, *J*(P,C) = 89.9 Hz), 81.9 (d, *J*(P,C) = 90.5 Hz), 78.3 (d, *J*(P,C) = 90.3 Hz), 78.2 (d, *J*(P,C) = 90.4 Hz), 73.8, 73.6, 73.5, 73.4, 72.9, 72.7, 72.6, 72.2, 71.5, 71.4, 71.2, 71.1, 36.3, 36.1, 34.9, 31.0, 21.5, 21.4, 21.3, 21.2 (CH_3) ppm. ^{31}P NMR (CD_2Cl_2, δ), 80.9, 80.7, 78.1 and 78.0 ppm. Mass spectrum (CI^+, m/z), 819 $[M+H]^+$. Accurate mass measurement [CI^+, m/z]: 818.9080 $[M+H]^+$, calculated mass for $C_{35}H_{32}Fe_2P_2S_6H$: 818.9081.

Compound 10a. Yellow paste (0.250 g, 33%). Selected IR (KBr, cm^{-1}): 1410(m), 1300(m), 1252(m), 1173(s), 1106(m), 1024(s), 1002(m), 825(s), 767(m), 732(m), 675(vs), 615(m), 535(s), 483(vs). ^{1}H NMR ($CDCl_3$, δ), 7.32-7.10 (m, 4H), 4.83-4.53 (m, 8H), 4.43 (s, 5H), 4.41 (s, 5H), 4.19-4.13 (m, 4H), 3.32-3.10 (m, 4H), 2.29-2.21 (m, 2H) ppm. ^{13}C NMR ($CDCl_3$, δ), 136.3 (d, *J*(P,C) = 66.7 Hz), 134.1 (d, *J*(P,C) = 10.4 Hz), 132.8, 131.2 (d, *J*(P,C) = 11.7 Hz), 127.9, 126.8, 78.3 (d, *J*(P,C) = 100.9 Hz), 72.1 (d, *J*(P,C) = 12.0 Hz), 71.4 (d, *J*(P,C) = 15.0 Hz), 71.0, 35.8, 32.1, 29.7 ppm. ^{31}P NMR ($CDCl_3$, δ), 79.4 ppm. Mass spectrum (EI^+, m/z), 770 $[M]^+$. Accurate mass measurement [EI^+, m/z]: 769.9004 $[M]^+$, calculated mass for $C_{31}H_{32}Fe_2P_2S_6$: 769.9002.

Compound 10b. Dark yellow paste (0.220 g, 29%). Selected IR (KBr, cm^{-1}): 1409(m), 1298(m), 1244(m), 1170(s), 1106(m), 1024(s), 1002(m), 908(s), 824(s), 768(m), 731(s), 674(s), 618(m), 532(s), 478(vs). ^{1}H NMR ($CDCl_3$, δ), 7.35-7.27 (m, 4H), 4.79-4.56 (m, 8H), 4.42 (s, 10H), 4.16-4.10 (m, 4H), 3.26-3.07 (m, 4H), 2.24-2.20 (m, 2H) ppm. ^{13}C NMR ($CDCl_3$, δ), 140.1, 134.1 (d, *J*(P,C) = 3.1 Hz), 131.6, 128.8, 78.5 (d, *J*(P,C) = 100.5 Hz), 72.0 (d, *J*(P,C) = 12.0 Hz), 71.5 (d, *J*(P,C) = 15.1 Hz), 71.0, 35.7, 32.5, 29.4 ppm. ^{31}P NMR ($CDCl_3$, δ), 79.5 ppm. Mass spectrum (EI^+, m/z), 770 $[M]^+$. Accurate mass measurement [EI^+, m/z]: 769.9004 $[M]^+$, calculated mass for $C_{31}H_{32}Fe_2P_2S_6$: 769.9002.

Compound 11. Dark yellow paste (0.402 g, 51%). Selected IR (KBr, cm^{-1}): 1455(m), 1414(m), 1172(s), 1105(m), 1024(s), 1002(m), 971(m), 908(m), 824(s), 765(m), 731(m), 674(s), 530(m), 479(s). ^{1}H NMR ($CDCl_3$, δ), 7.42-7.19 (m, 4H), 4.68-4.50 (m, 8H), 4.39 (s, 10H), 3.99-3.84 (m, 4H), 3.43-3.39 (m, 4H), 1.97-1.84 (m, 4H) ppm. ^{13}C NMR ($CDCl_3$, δ), 135.4, 131.2, 128.3, 80.1 (d, *J*(P,C) = 96.0 Hz), 71.8 (d, *J*(P,C) = 11.8 Hz), 71.4 (d, *J*(P,C) = 14.9 Hz), 70.8, 35.2, 32.9, 28.6 ppm. ^{31}P NMR ($CDCl_3$, δ), 79.5 ppm. Mass spectrum (EI^+, m/z), 784 $[M]^+$. Accurate mass measurement [EI^+, m/z]: 783.9156 $[M]^+$, calculated mass for $C_{32}H_{34}Fe_2P_2S_6$: 783.9159.

Compound 12a. Pale yellow paste (0.200 g, 25%). Selected IR (KBr, cm^{-1}): 1489(m), 1453(m), 1410(m), 1171(s), 1106(m), 1024(s), 908(m), 824(s), 766(m), 731(s), 676(vs), 618(m), 530(s), 479(vs), 259(s). ^{1}H NMR ($CDCl_3$, δ), 7.32-7.17 (m, 4H), 4.73-4.50 (m, 8H), 4.39 (s, 10H), 4.36-4.16 (m, 4H), 3.94-3.83 (m, 4H), 3.23-2.97 (m, 4H), 1.90-1.55 (m, 4H) ppm. ^{13}C NMR ($CDCl_3$, δ), 138.7, 135.0 (d, *J*(P,C) = 3.1 Hz), 130.9, 128.3, 79.7 (d, *J*(P,C) = 100.9 Hz), 71.8 (d, *J*(P,C) = 12.5 Hz), 71.4 (d, *J*(P,C) = 15.5 Hz), 70.8, 35.5, 33.2, 28.8, 26.9

ppm. ^{31}P NMR ($CDCl_3$, δ), 81.8 ppm. Mass spectrum (EI^+, m/z), 812 $[M]^+$. Accurate mass measurement [EI^+, m/z]: 811.9468 $[M]^+$, calculated mass for $C_{34}H_{38}Fe_2P_2S_6$: 811.9471.

Compound 12b. Pale yellow paste (0.250 g, 31%). Two diastereomers were found in *ca.* 3:2 intensity ratio. Selected IR (KBr, cm^{-1}): 1489(m), 1453(m), 1410(m), 1385(m), 1170(s), 1106(m), 1024(s), 1001(m), 823(s), 765(m), 676(vs), 618(m), 529(m), 478(vs), 259(s). ^{1}H NMR ($CDCl_3$, δ), 7.37-7.19 (m, 8H), 4.75-4.52 (m, 16H), 4.40 (s, 10H), 4.39 (s, 10H), 4.36-4.17 (m, 8H), 3.93-3.84 (m, 8H), 3.21-2.90 (m, 8H), 1.94-1.29 (m, 8H) ppm. ^{13}C NMR ($CDCl_3$, δ), 140.2, 135.2 (d, J(P,C) = 3.1 Hz), 133.7 (d, J(P,C) = 3.1 Hz), 130.9, 129.8, 128.5, 127.7, 80.1 (d, J(P,C) = 100.6 Hz), 79.8 (d, J(P,C) = 101.1 Hz), 71.9, 71.7, 71.5, 71.3, 70.9, 70.8, 35.5, 35.2, 33.6, 33.5, 29.9, 28.8, 27.0, 26.1 ppm. ^{31}P NMR ($CDCl_3$, δ), 81.7 and 80.5 ppm. Mass spectrum (EI^+, m/z), 812 $[M]^+$. Accurate mass measurement [EI^+, m/z]: 811.9467 $[M]^+$, calculated mass for $C_{34}H_{38}Fe_2P_2S_6$: 811.9471.

Compound 14. Pale-yellow solid (0.080 g, 10%). Selected IR (KBr, cm^{-1}): 1510(m), 1407(m), 1242(m), 1196(s), 1150(m), 1104(m), 1023(s), 891(m), 844(m), 810(s), 768(m), 656(m), 632(m), 560(s), 501(s), 477(s). ^{1}H NMR ($CDCl_3$, δ), 7.34-7.18 (m, 8H), 4.68-4.54 (m, 8H), 4.40 (s, 10H), 4.38-4.34 (m, 4H), 3.91-3.84 (m, 4H) ppm. ^{13}C NMR ($CDCl_3$, δ), 136.8, 129.1, 78.5 (d, J(P,C) = 92.6 Hz), 72.1 (d, J(P,C) = 12.5 Hz), 71.8 (d, J(P,C) = 15.6 Hz), 70.5, 35.0 ppm. ^{31}P NMR ($CDCl_3$, δ), 59.8 ppm. Mass spectrum (CI^+, m/z), 801 $[M+H]^+$. Accurate mass measurement [CI^+, m/z]: 800.9690 $[M+H]^+$, calculated mass for $C_{36}H_{34}Fe_2O_2S_4H$: 800.9694.

3. Details of the X-ray Data Collections and Refinements for 10a, 10b, 11, 12a, 12b and 14

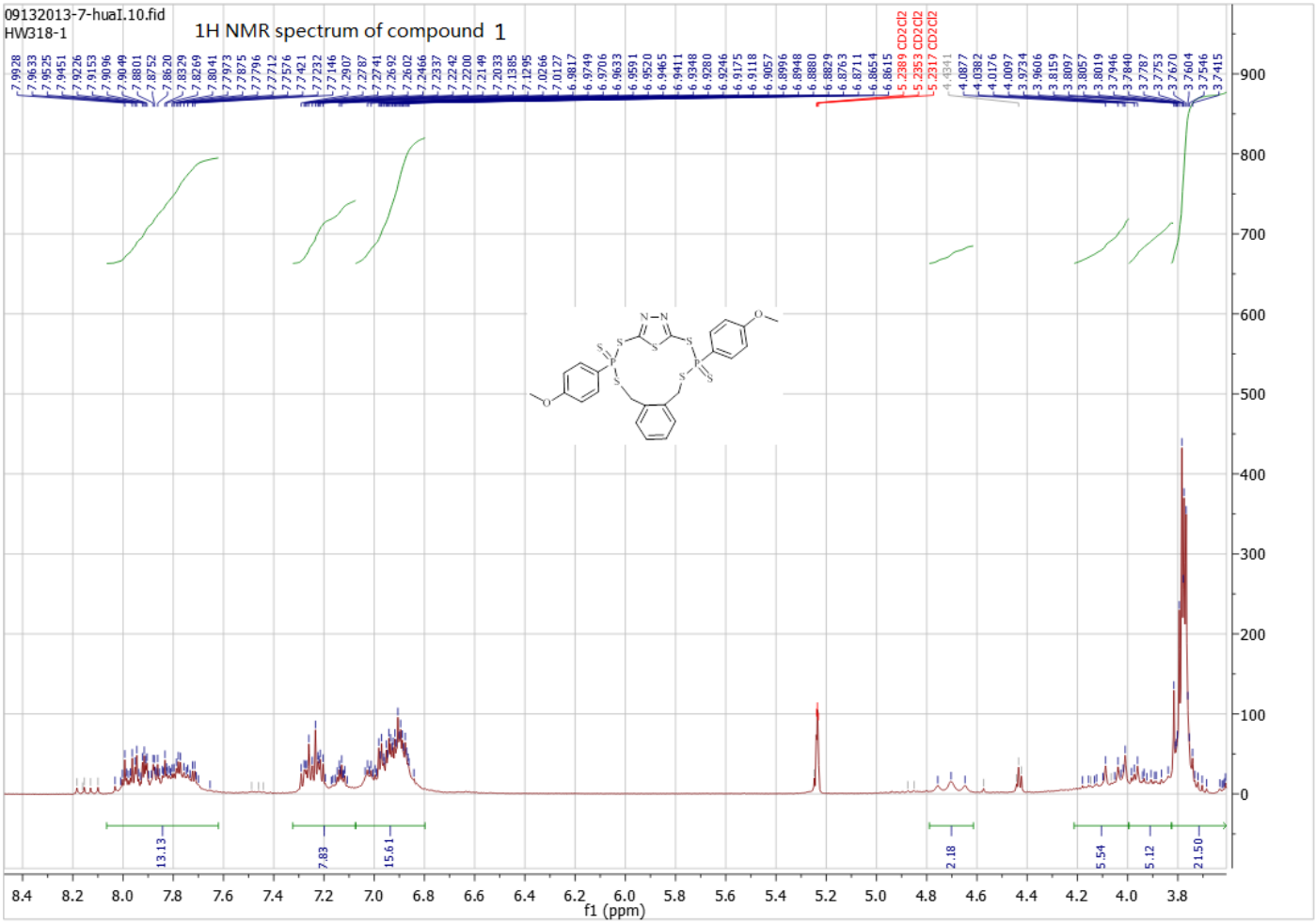
Table S1. Details of the X-ray data collections and refinements for **10a**, **10b** and **11**

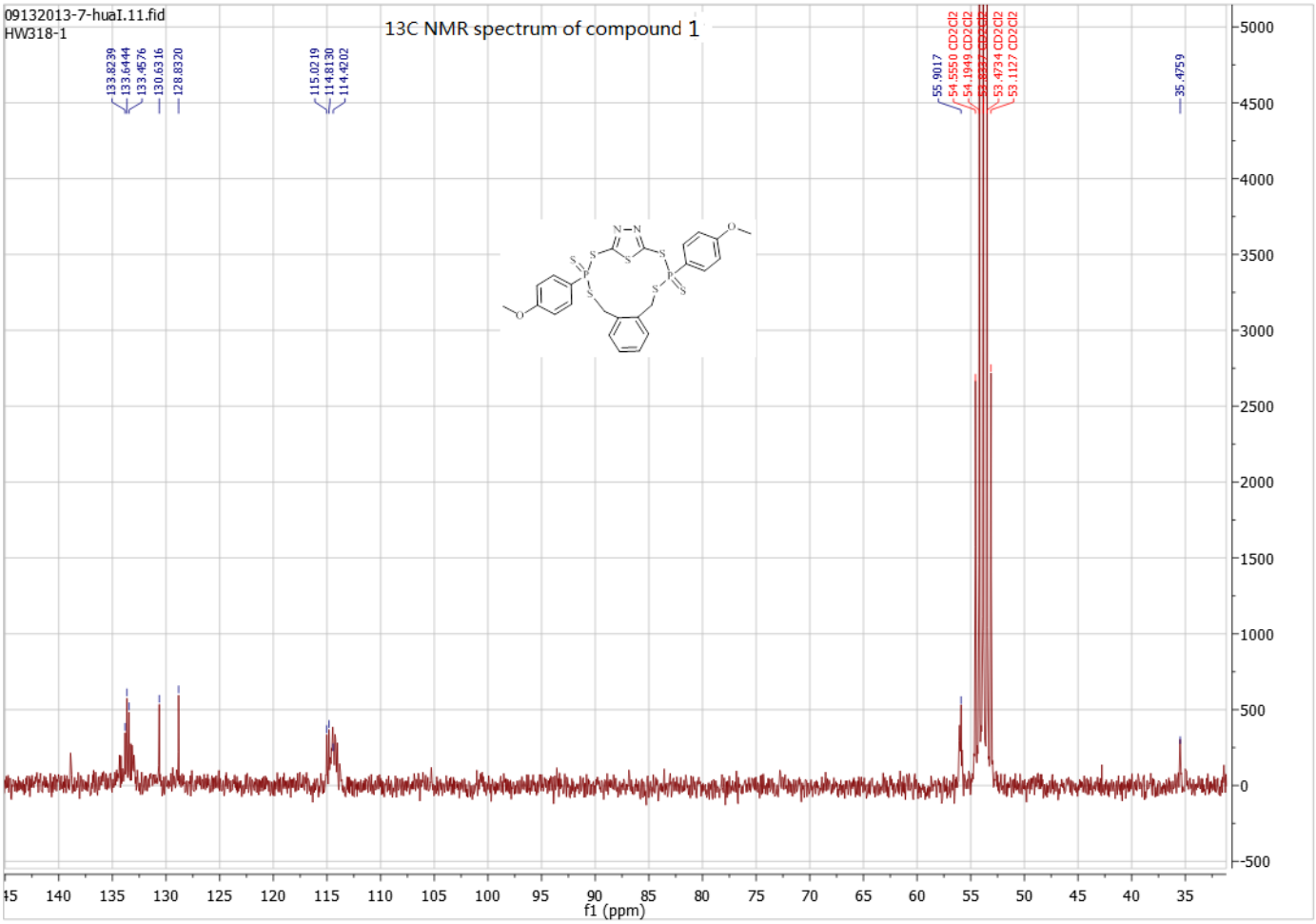
Compound	**10a**	**10b**	**11**
Formula	$C_{31}H_{32}Fe_2P_2S_6$	$C_{31}H_{32}Fe_2P_2S_6$	$C_{32}H_{34}Fe_2P_2S_6$
M	770.60	770.60	784.62
Crystal system	monoclinic	monoclinic	monoclinic
Space group	*C2/c*	*Cc*	$P2_1/c$
a/Å	8.3844(16)	12.3542(13)	17.762(3)
b/Å	12.371(2)	8.3008(9)	12.2131(17)
c/Å	30.530(5)	31.047(3)	16.275(2)
A	90	90	90
B	92.731(5)	93.806(3)	115.073(3)
Γ	90	90	90
U/A^3	3163.1(9)	3176.8(6)	3197.8(8)
Z	4	4	4
μ/cm^{-1}	14.366	14.303	14.225
Reflections collected	17845	31053	38576
Independent reflections	2681	5736	5859
R_{int}	0.0527	0.0486	0.0381
R1	0.0939	0.0482	0.0393
wR2 [I > 2σ(I)]	0.2339	0.1234	0.0995

Table S2. Details of the X-ray data collections and refinements for **12a**, **12b** and **14**

Compound	**12a**	**12b**	**14**
Formula	$C_{34}H_{38}Fe_2P_2S_6$	$C_{34}H_{38}Fe_2P_2S_6$	$C_{36}H_{34}Fe_2O_2P_2S_4$
M	812.68	812.68	800.54
Crystal system	monoclinic	monoclinic	monoclinic
Space group	$P2_1/c$	$C2$	$P2_1/c$
a/Å	7.5941(7)	27.688(4)	19.112(6)
b/Å	24.3854(18)	7.5726(8)	11.516(4)
c/Å	19.0840(19)	34.772(6)	7.491(3)
A	90	90	90
B	91.056(3)	106.834(4)	95.025(8)
Γ	90	90	90
U/A^3	3533.5(5)	6978.2(17)	1624.4(10)
Z	4	8	2
μ/cm^{-1}	12.903	13.067	12.689
Reflections collected	43237	42175	30856
Independent reflections	6472	12617	2989
R_{int}	0.0332	0.0785	0.1499
R1	0.0435	0.0480	0.0853
wR2 [I > 2σ(I)]	0.1080	0.0890	0.2371

4. ^{1}H and ^{13}C NMR Spectra of Compounds 1-3, 5, 6, 8, 9, 10a, 10b, 11, 12a, 12b and 14

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114.4202
55.9017
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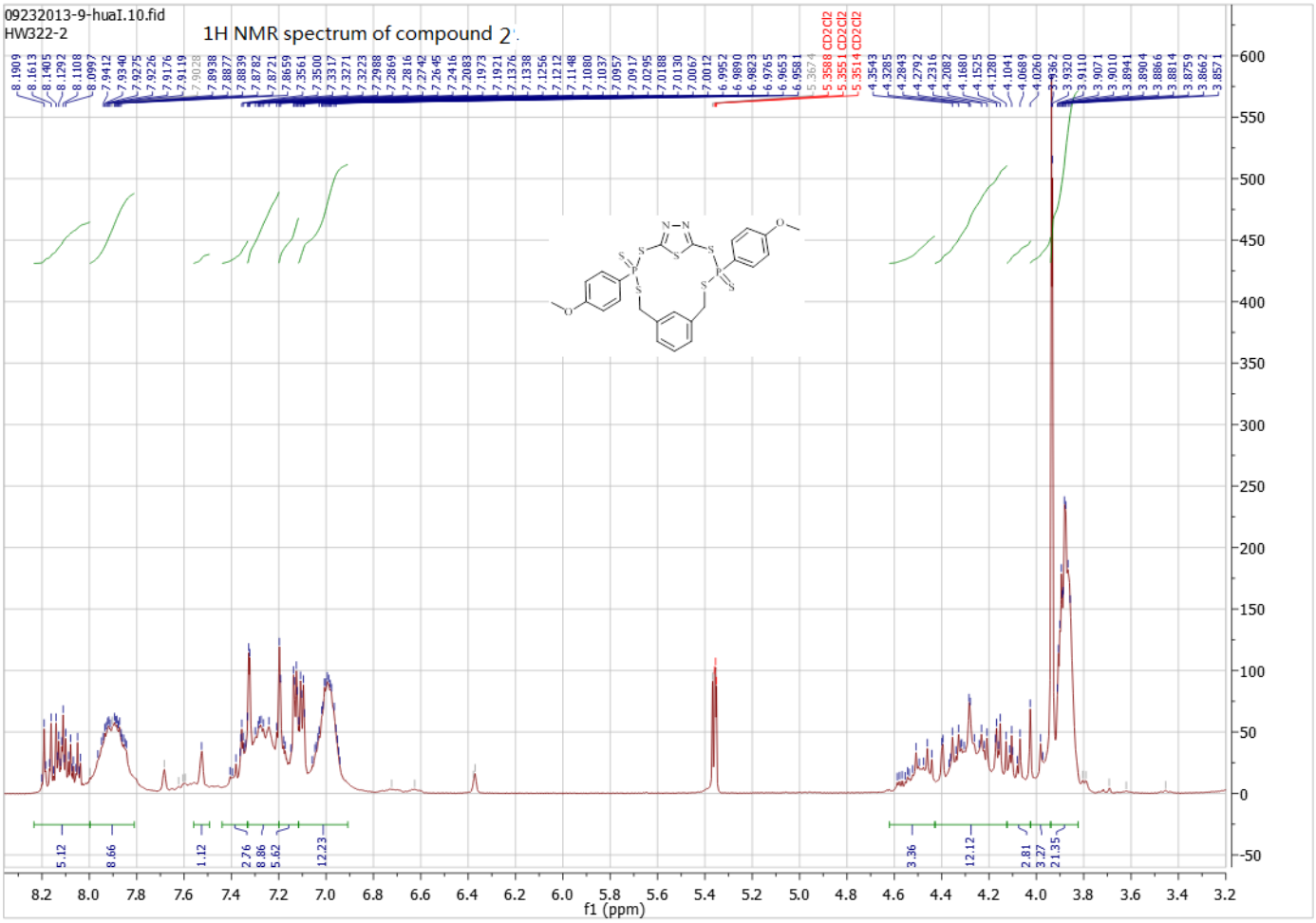
09232013-9-huaI.10.fid
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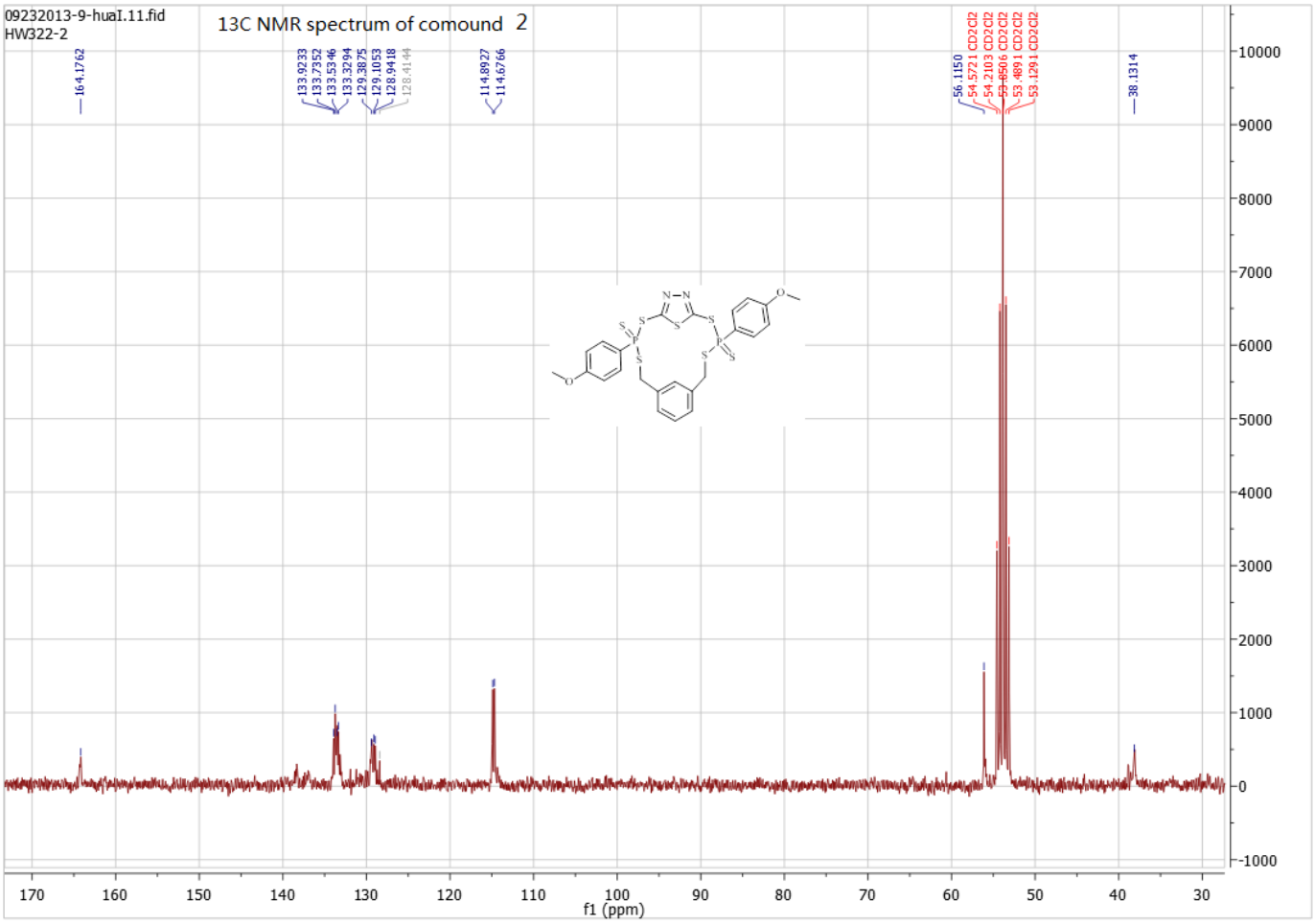
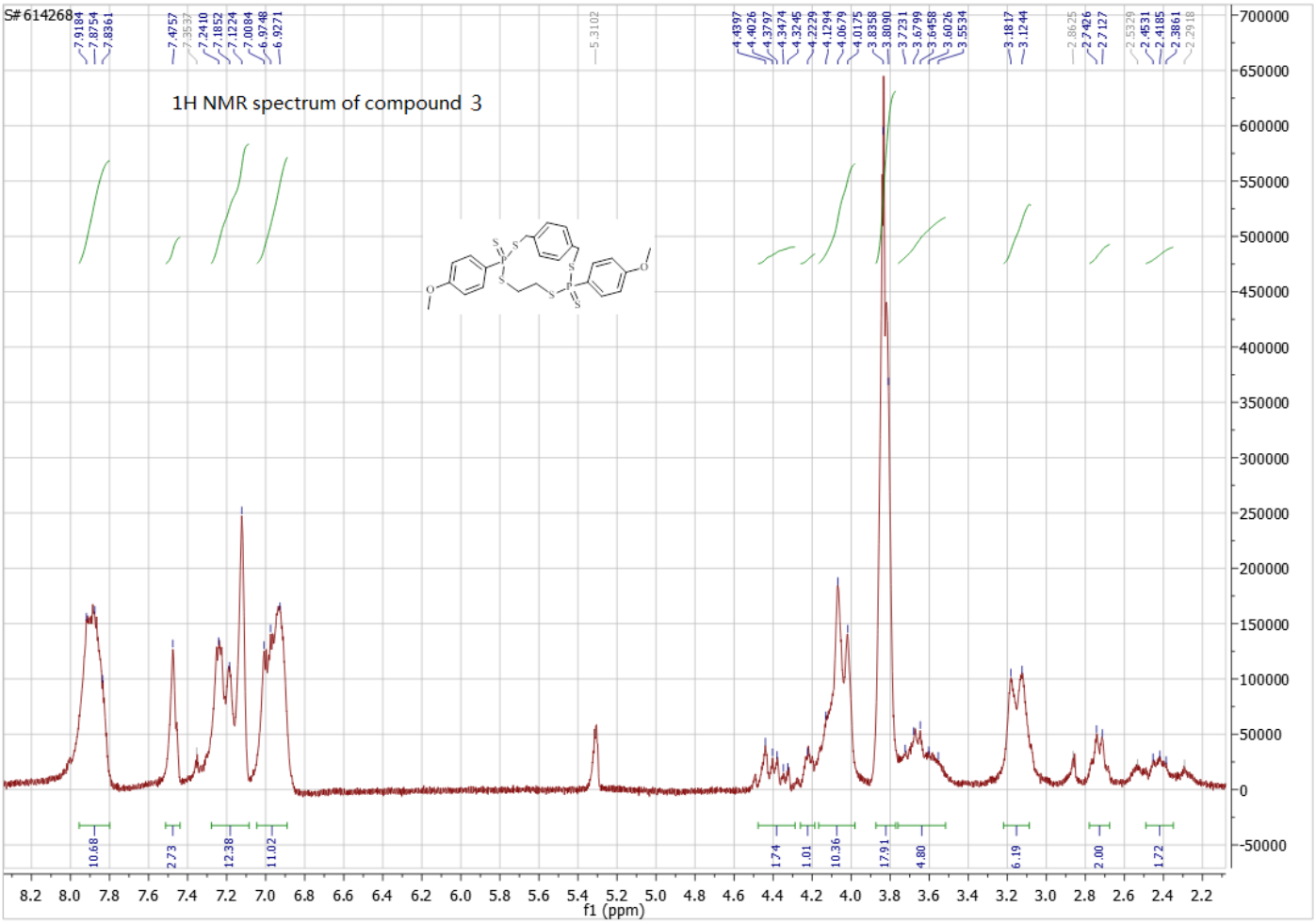
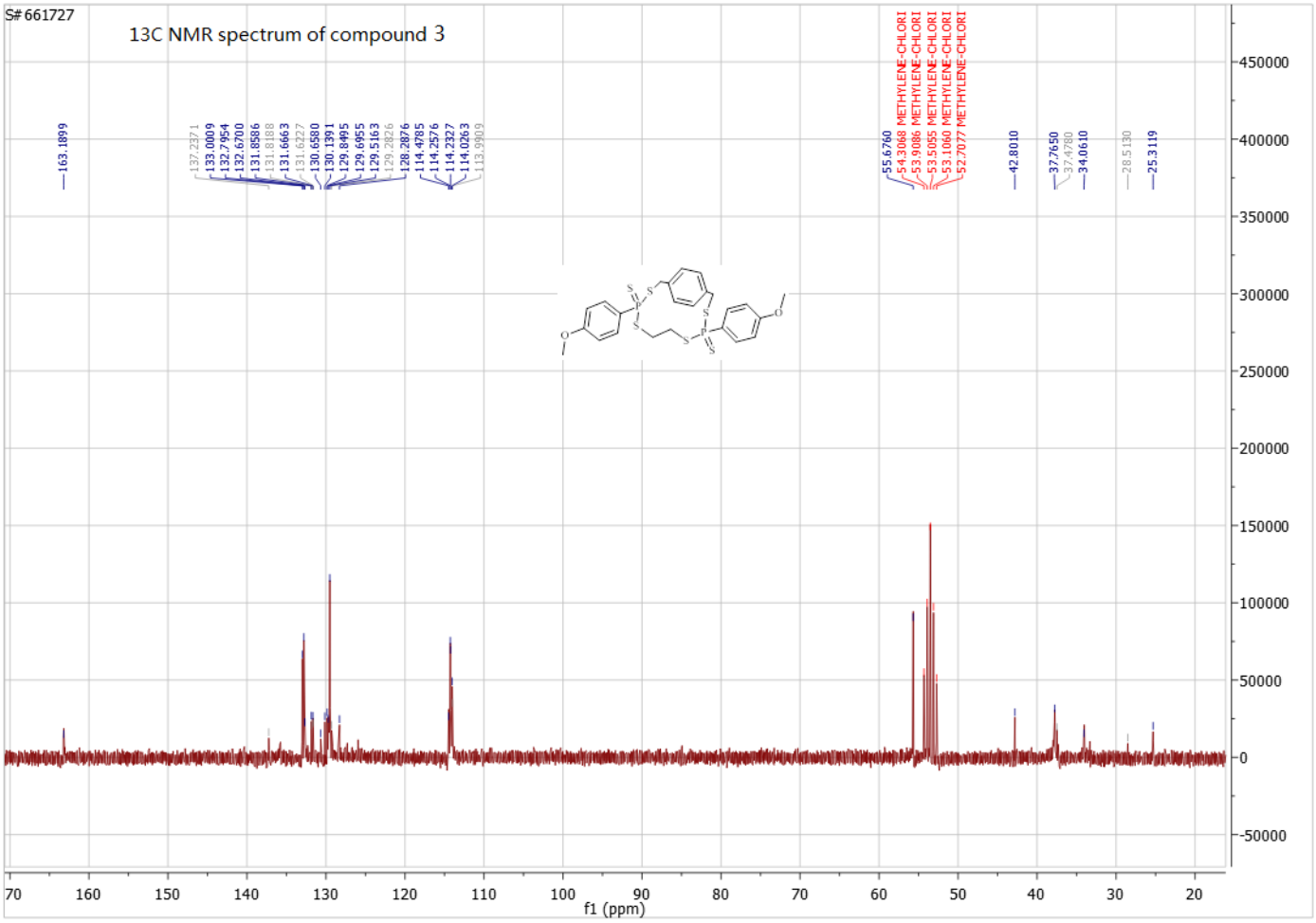
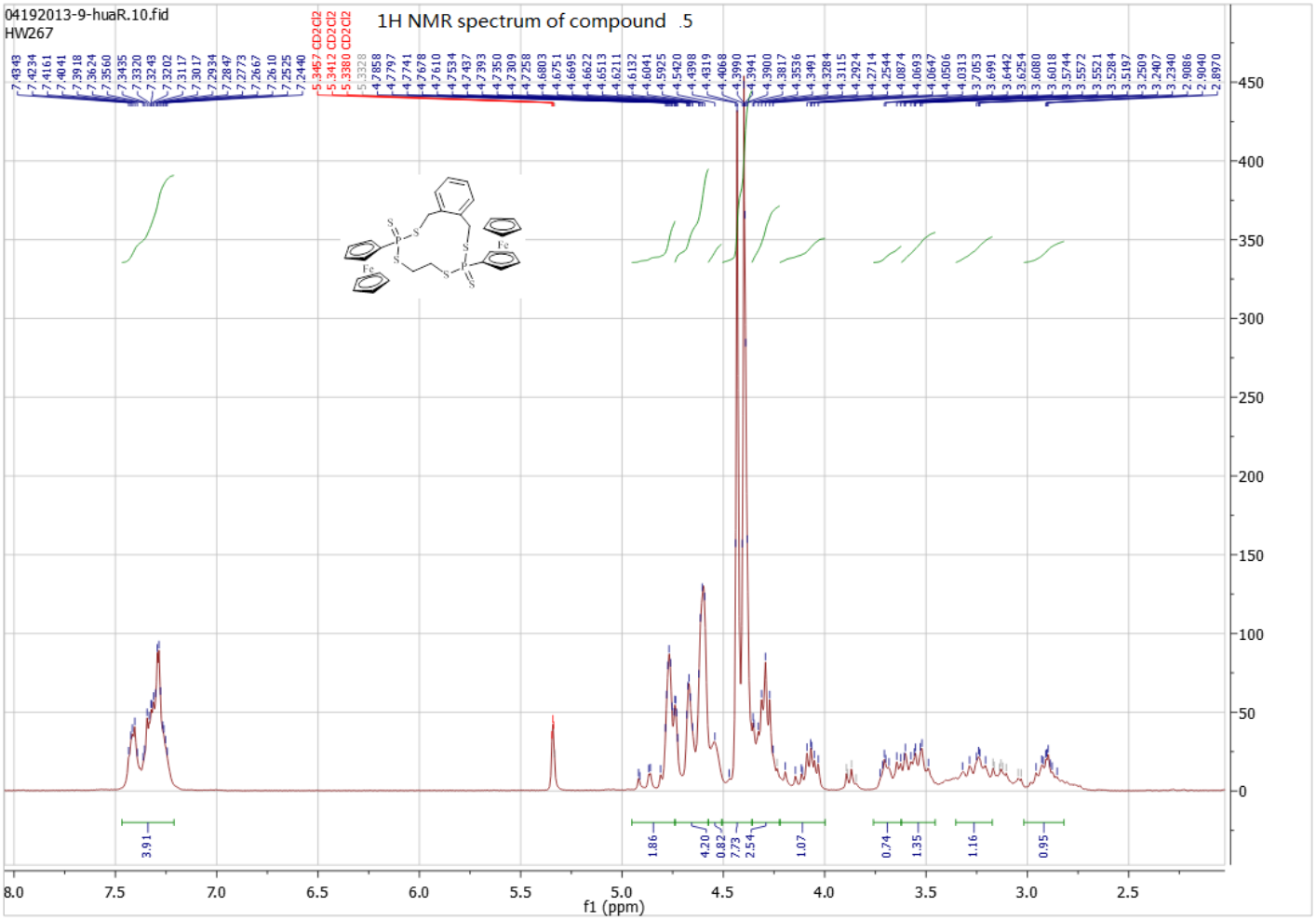
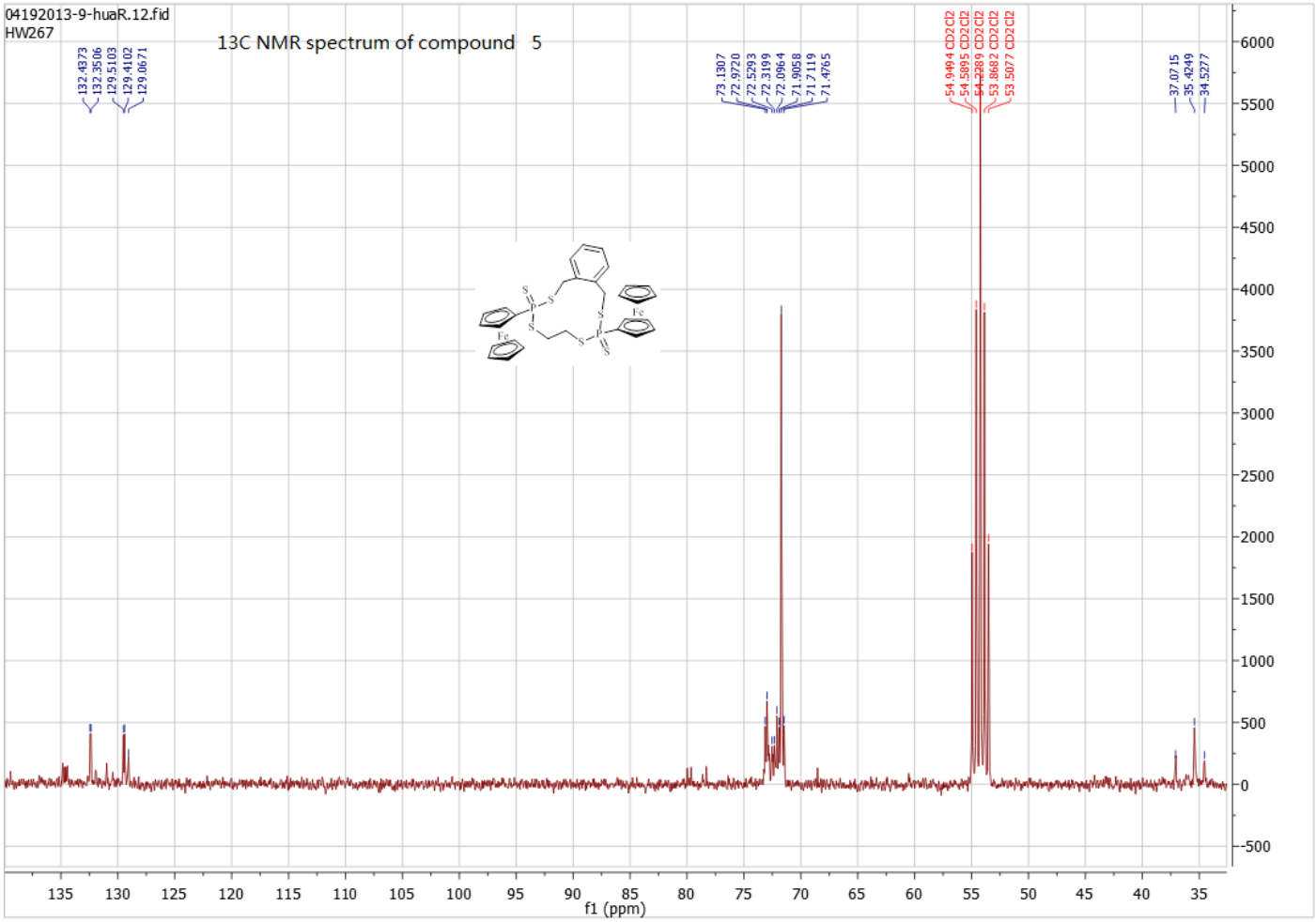
1H NMR spectrum of compound 2
f1 (ppm)

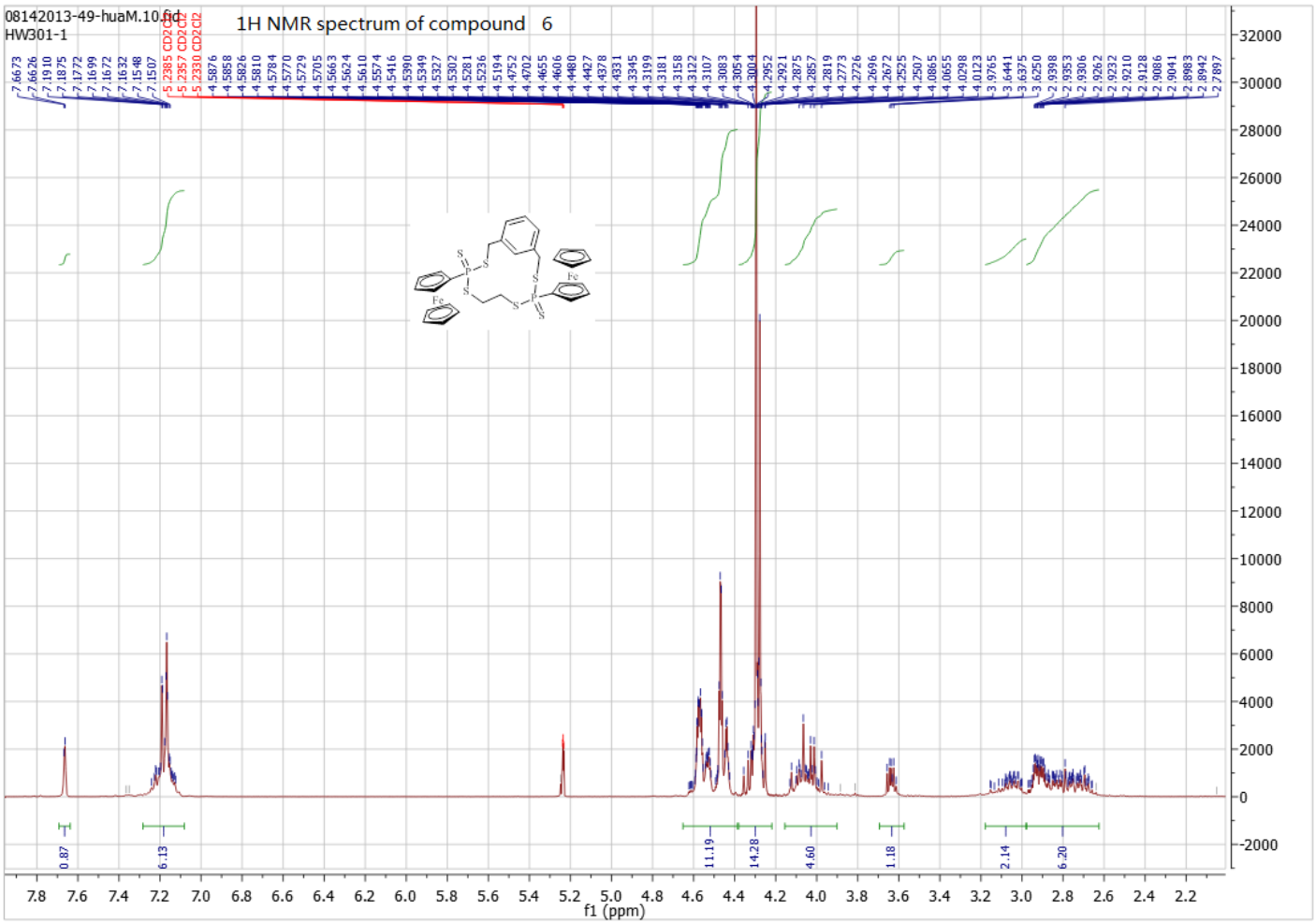

09232013-9-huaI.11.fid
HW322-2
13C NMR spectrum of comound 2
164.1762
133.9233
133.7352
133.5546
133.3294
129.3875
129.1053
128.9418
128.4144
114.8927
114.6766
56.1150
54.5721 CD2Cl2
54.2103 CD2Cl2
53.8506 CD2Cl2
53.4891 CD2Cl2
53.1291 CD2Cl2
38.1314
f1 (ppm)

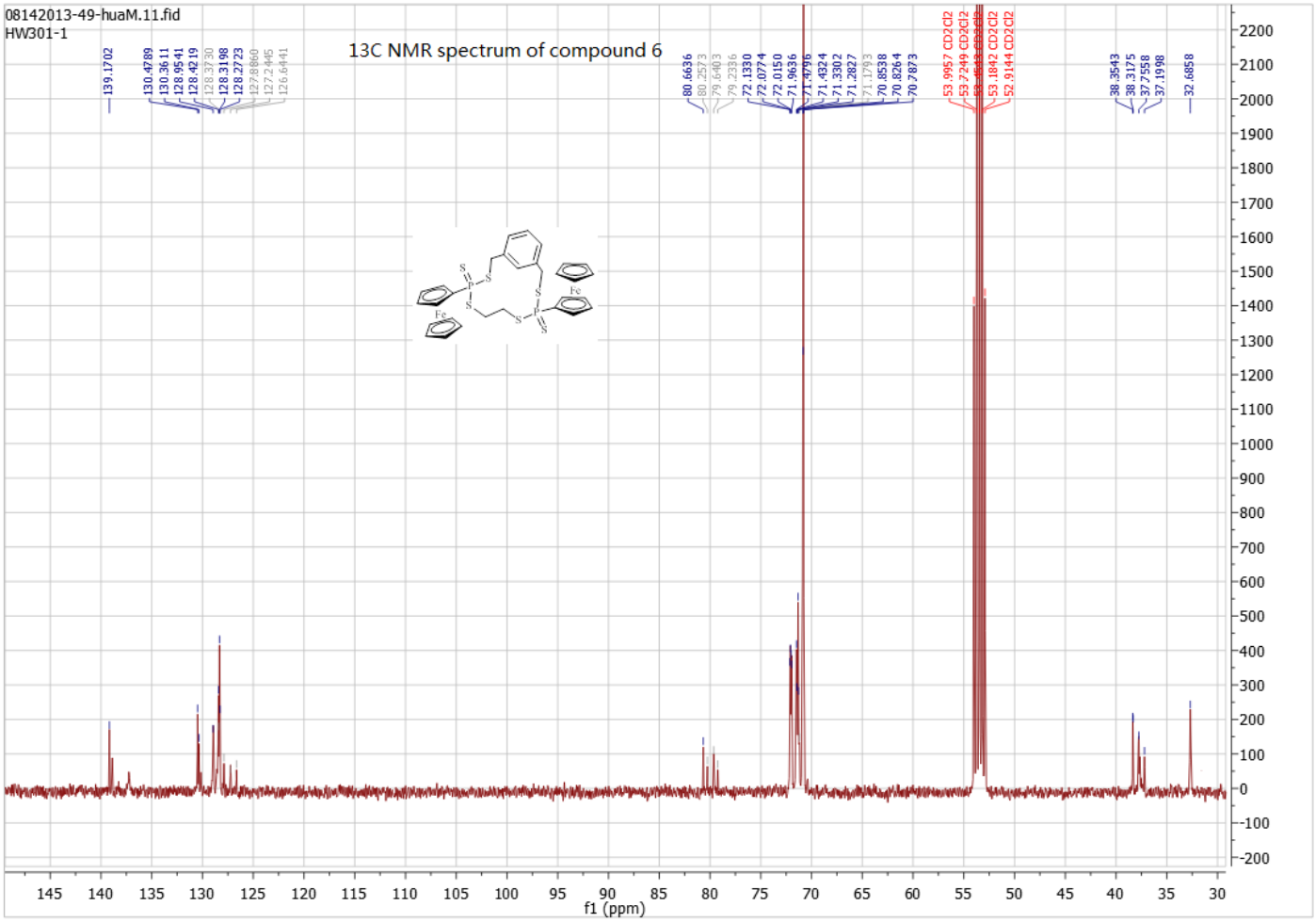

S# 614268
1H NMR spectrum of compound 3
f1 (ppm)

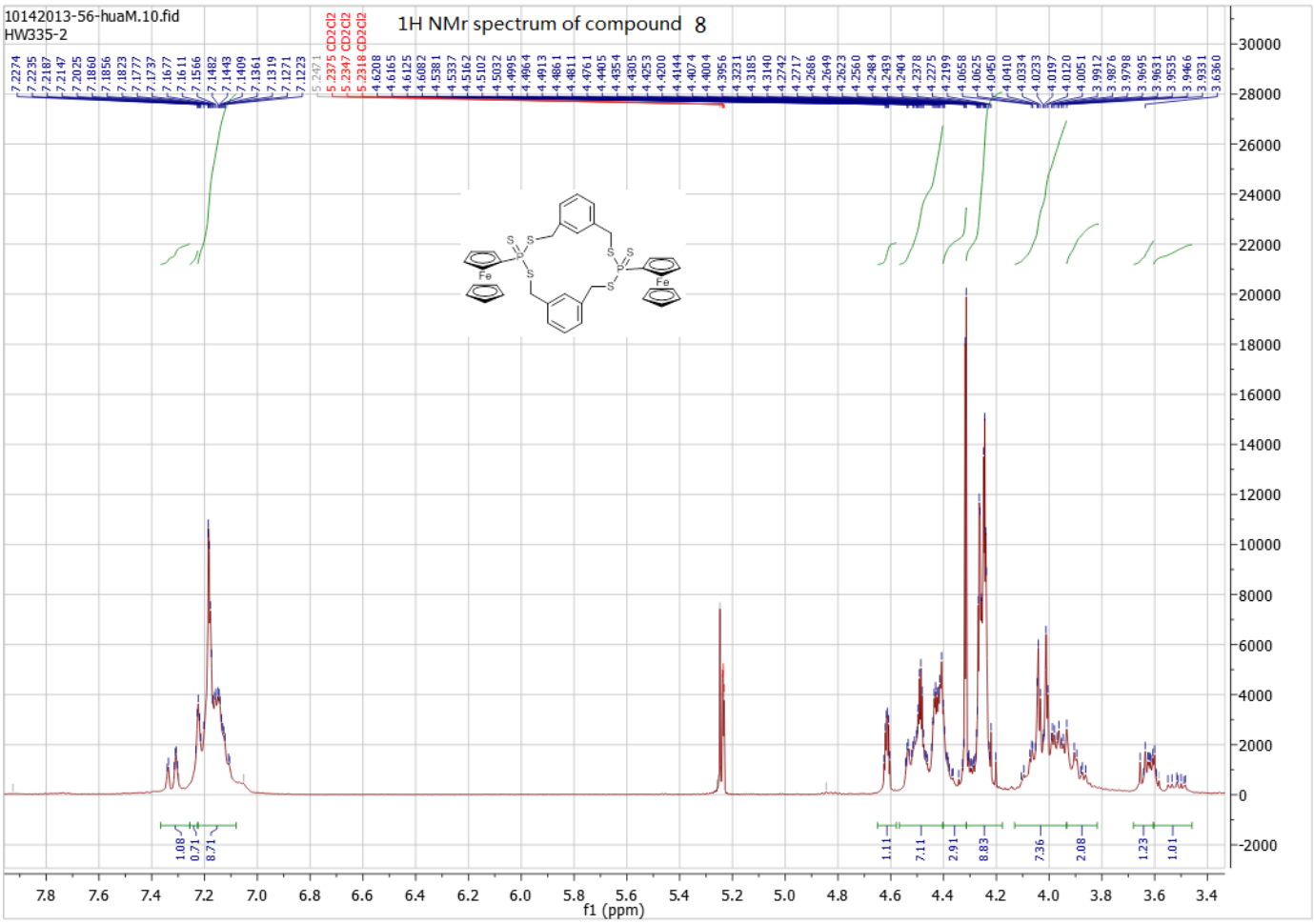

S# 661727
13C NMR spectrum of compound 3
163.1899
137.2371
133.0009
132.7954
132.6700
131.8586
131.8188
131.6663
131.6227
130.6580
130.1391
129.8495
129.6955
129.5163
129.2826
128.2876
114.4785
114.2576
114.2327
114.0263
113.9909
55.6760 METHYLENE-CHLORI
54.3068 METHYLENE-CHLORI
53.9086 METHYLENE-CHLORI
53.5055 METHYLENE-CHLORI
53.1060 METHYLENE-CHLORI
52.7077 METHYLENE-CHLORI
42.8010
37.7650
37.4780
34.0610
28.5130
25.3119
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
70
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

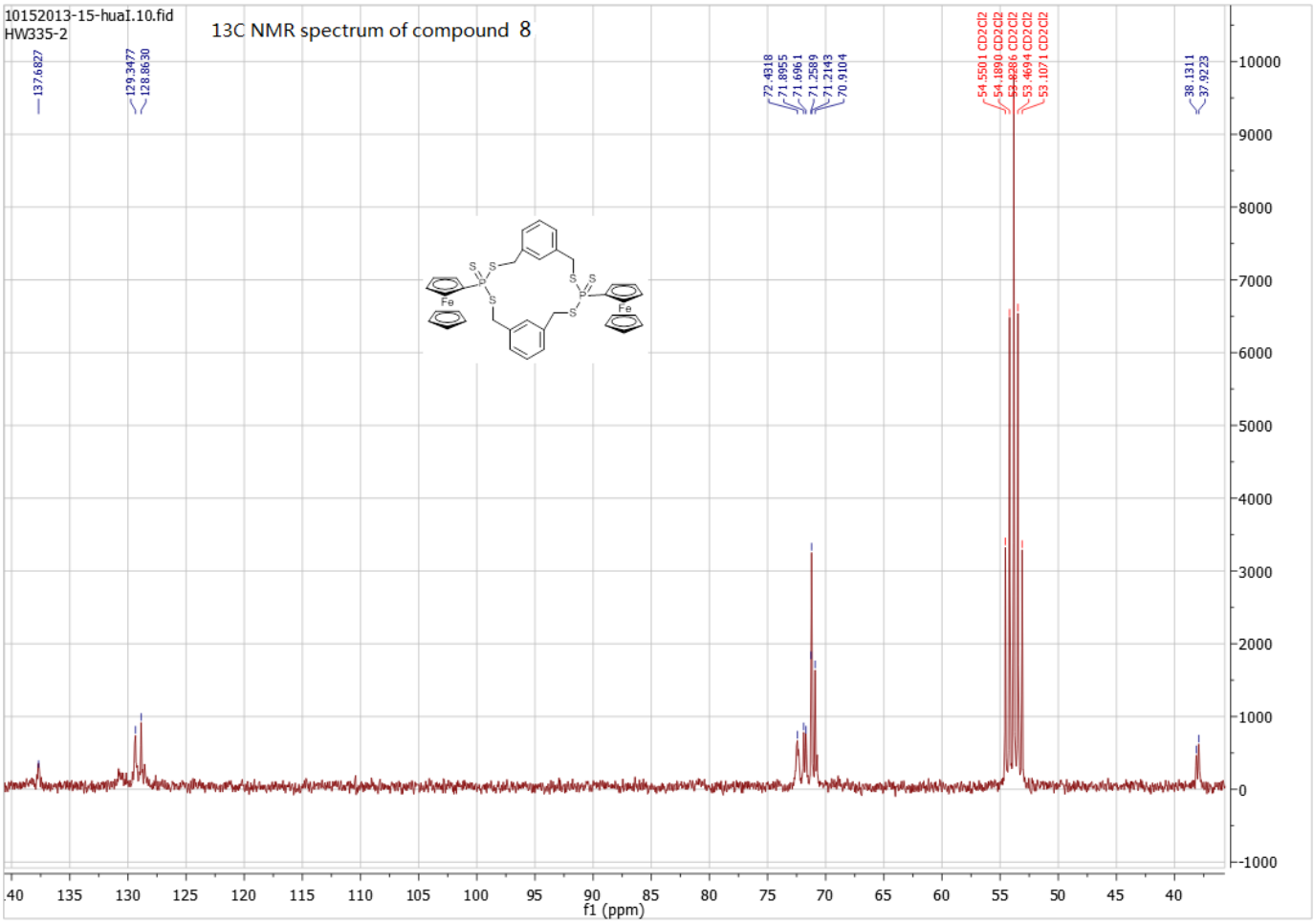

04192013-9-huaR.10.fid
HW267
1H NMR spectrum of compound .5
f1 (ppm)

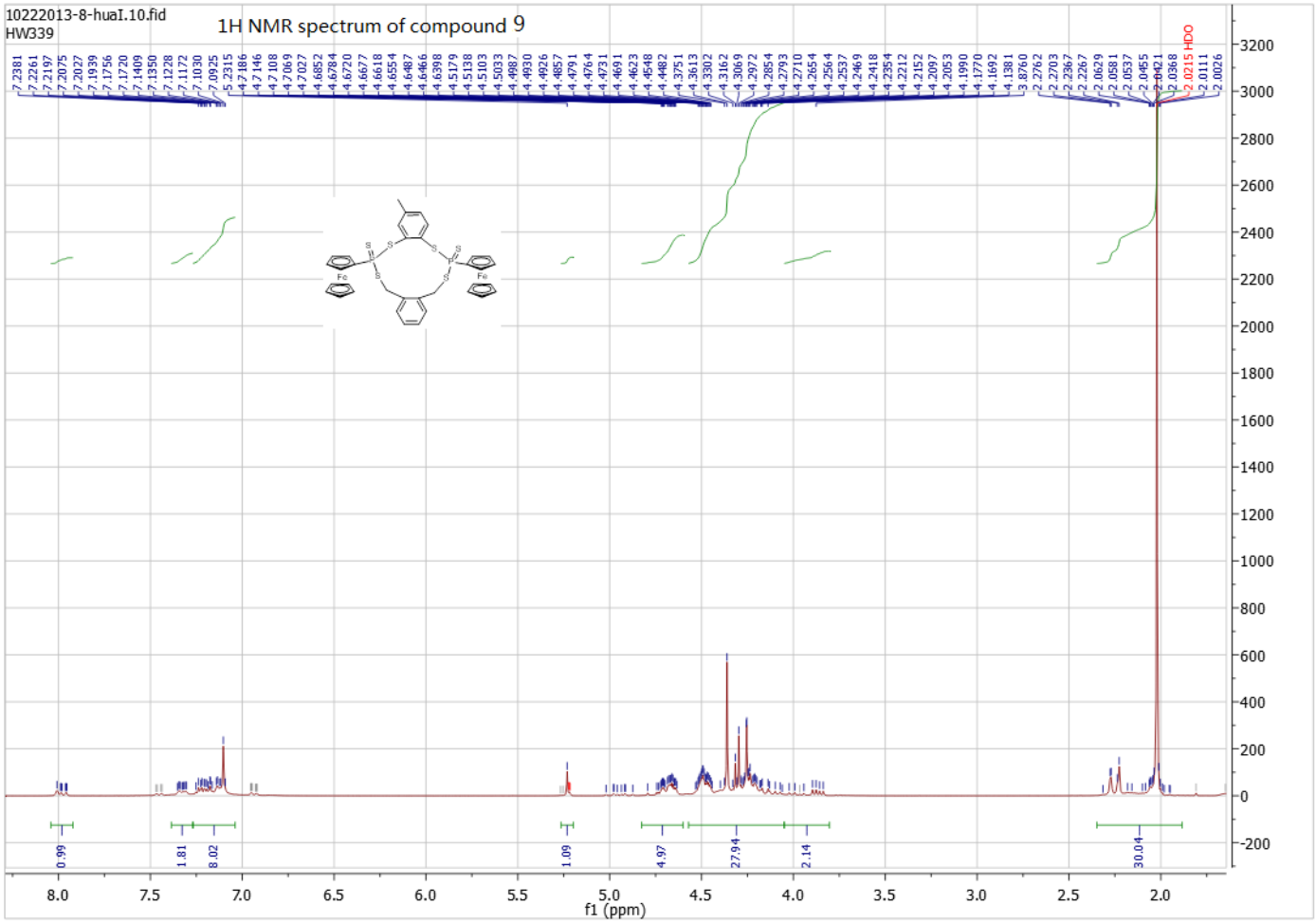

04192013-9-huaR.12.fid
HW267
13C NMR spectrum of compound 5
132.4373
132.3506
129.5103
129.4102
129.0671
73.1307
72.9720
72.5293
72.3199
72.0964
71.9058
71.7119
71.4765
54.9494 CD2Cl2
54.5895 CD2Cl2
54.2289 CD2Cl2
53.8682 CD2Cl2
53.5077 CD2Cl2
37.0715
35.4249
34.5277
Fe
Fe
f1 (ppm)

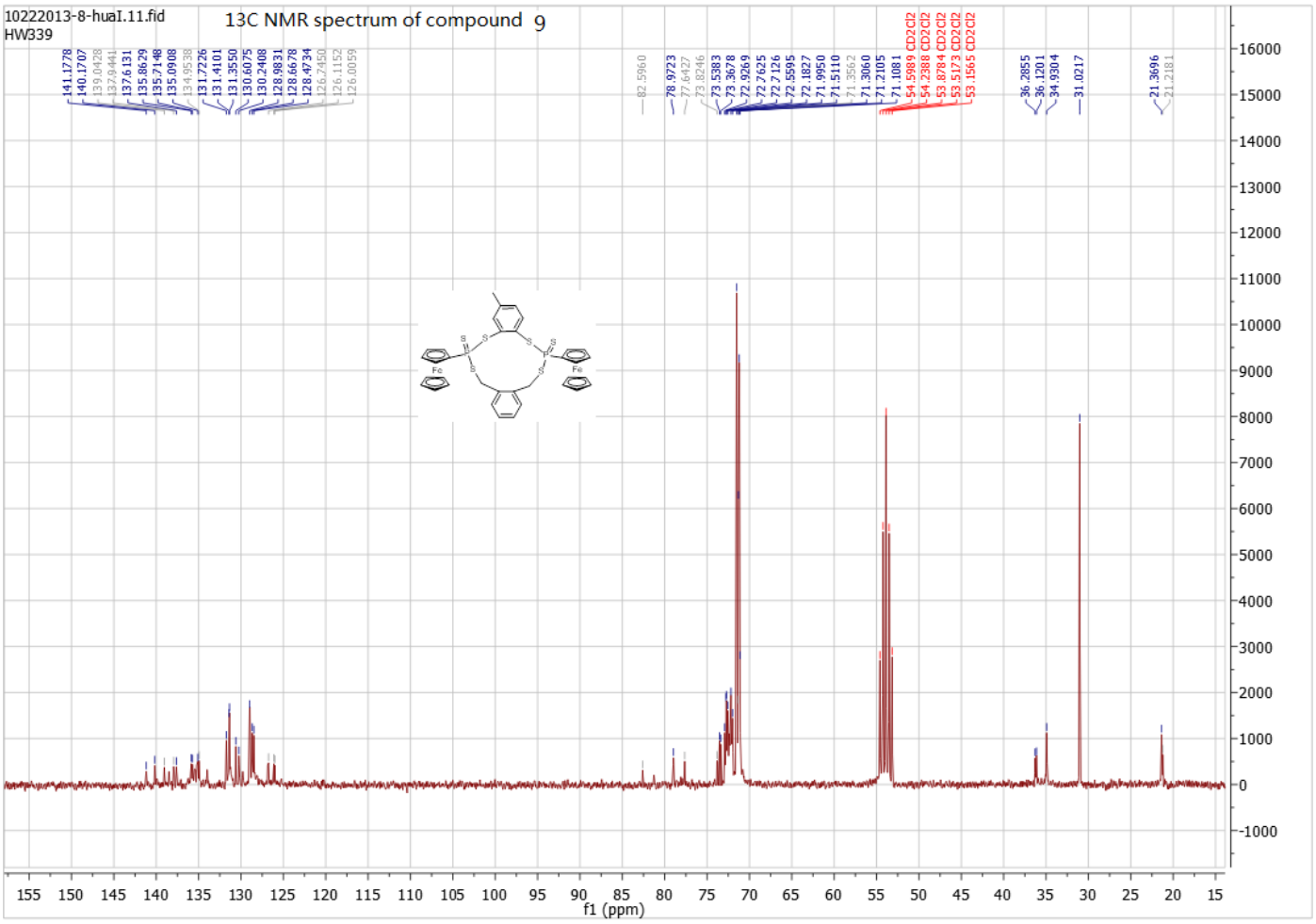

08142013-49-huaM.10.fid
HW301-1
1H NMR spectrum of compound 6
f1 (ppm)

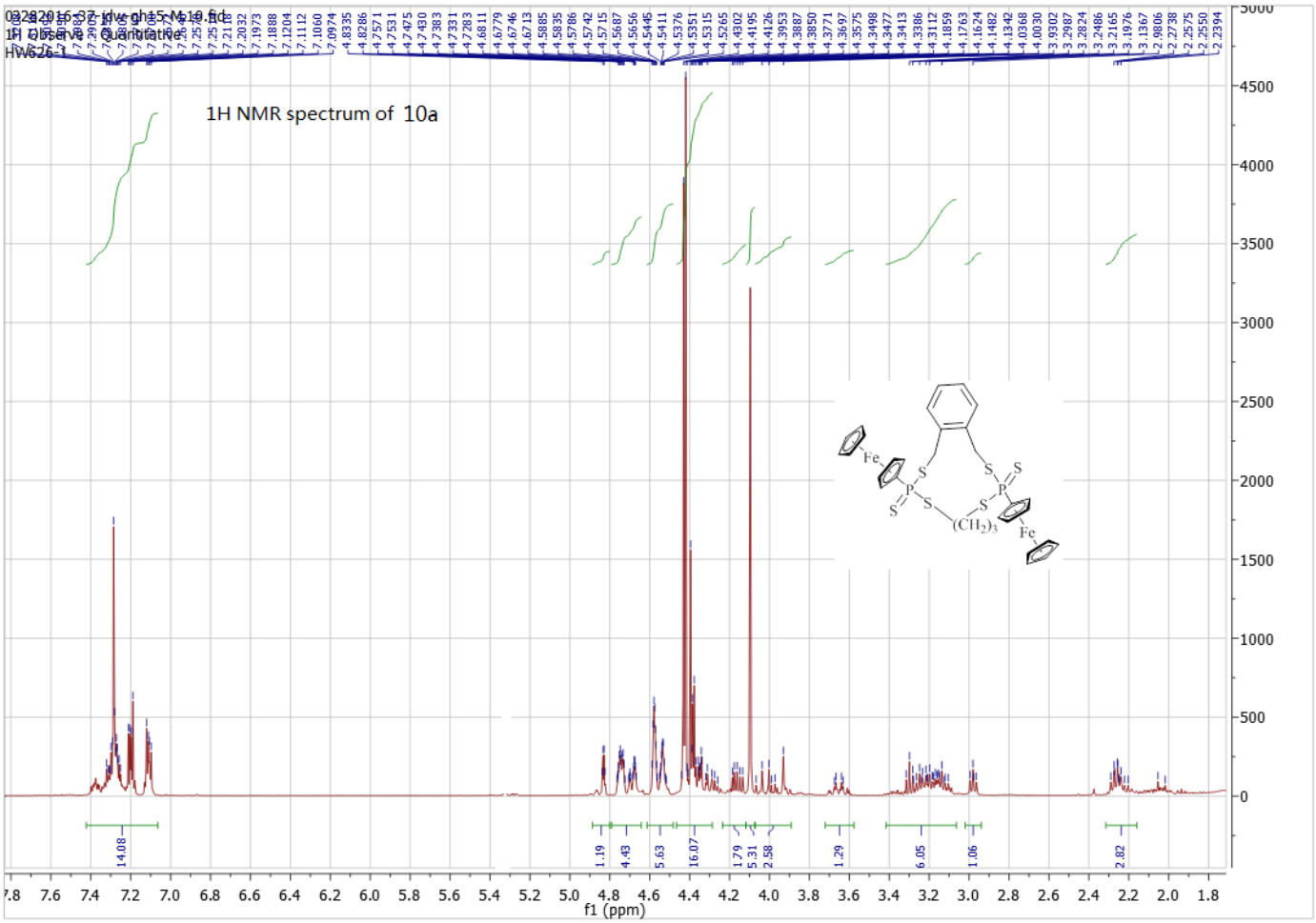

08142013-49-huaM.11.fid
HW301-1
13C NMR spectrum of compound 6
f1 (ppm)

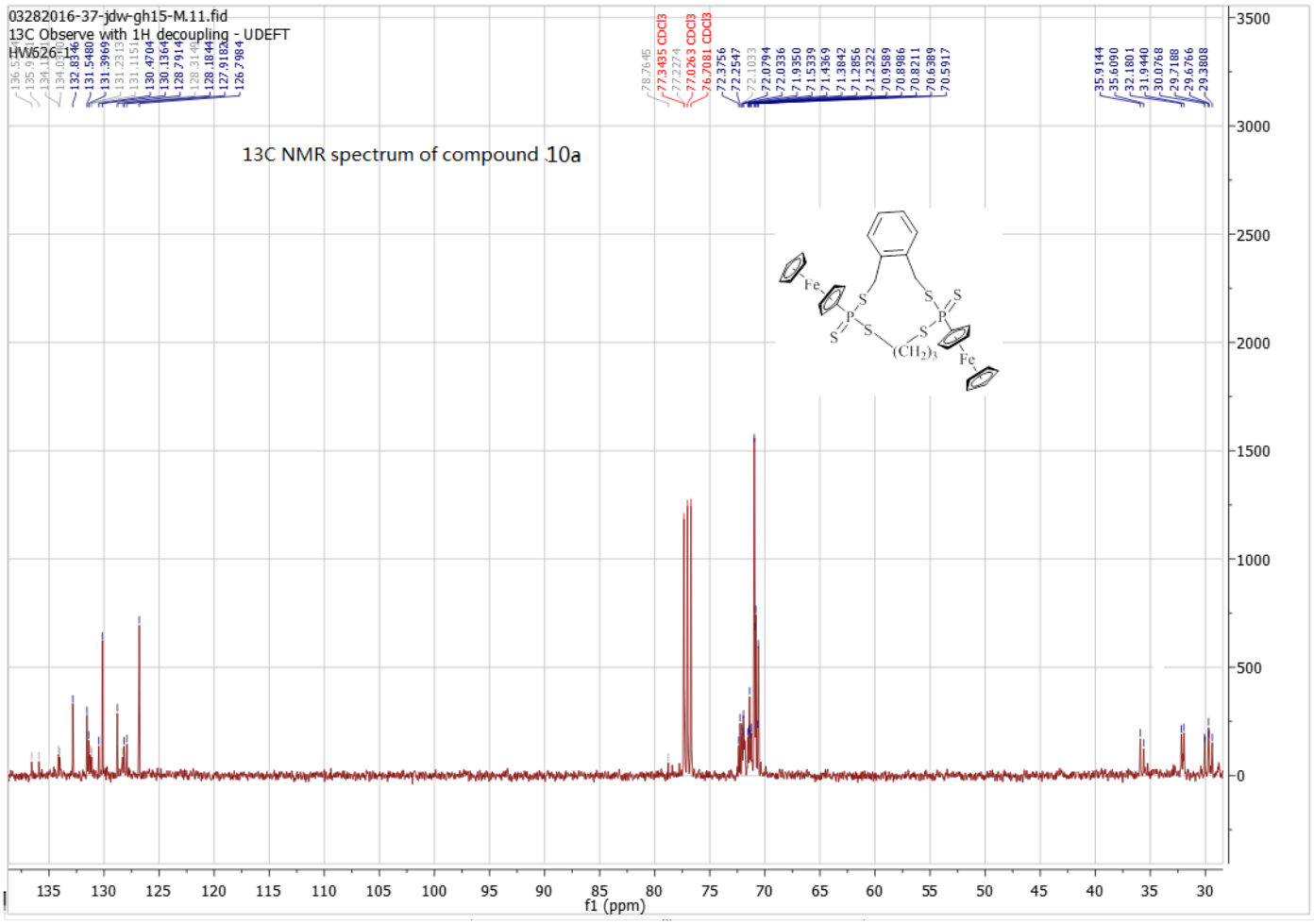

10142013-56-huaM.10.fid
HW335-2
1H NMr spectrum of compound 8
f1 (ppm)

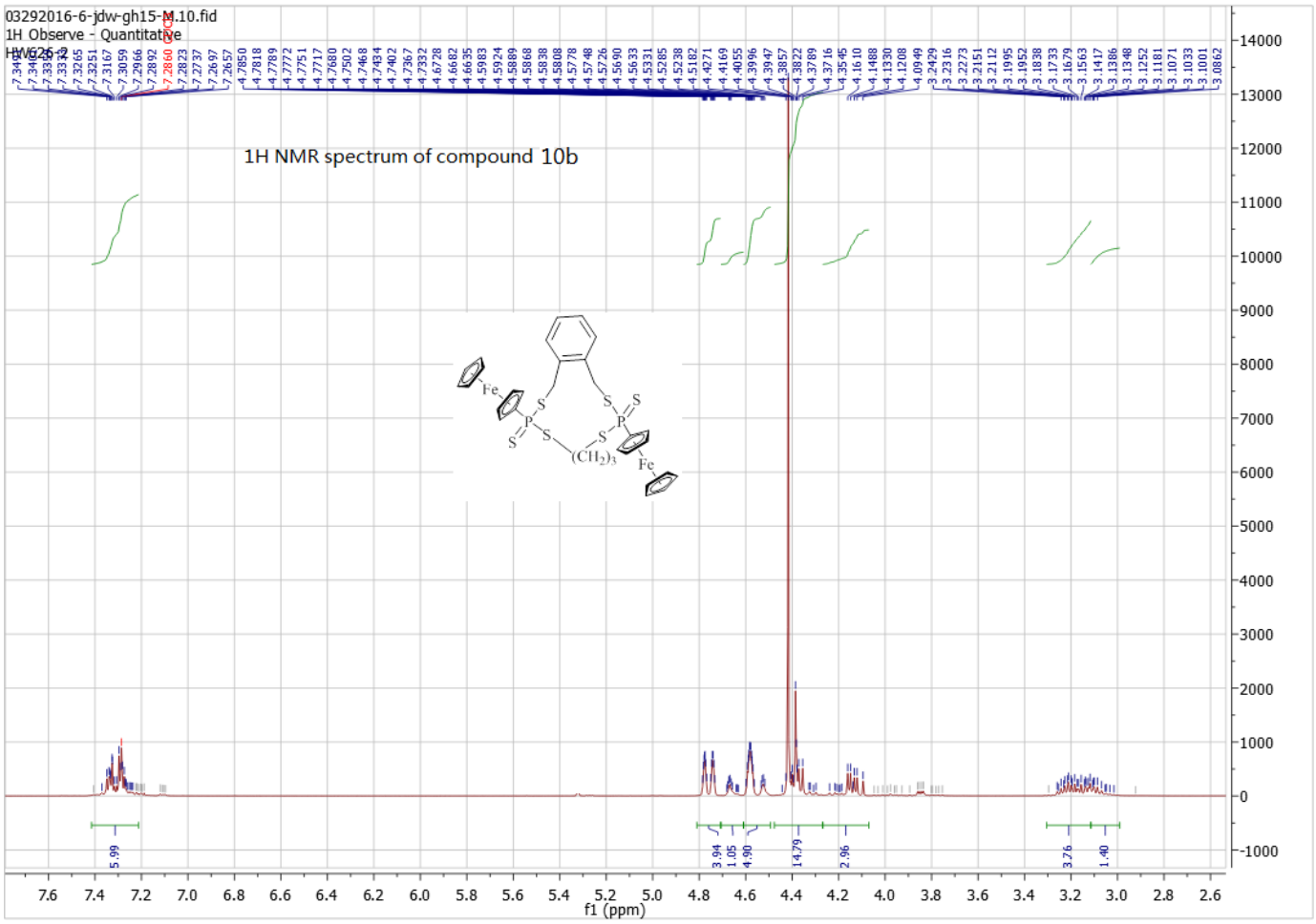

10152013-15-huaI.10.fid
HW335-2
13C NMR spectrum of compound 8
137.6827
129.3477
128.8630
72.4318
71.8955
71.6961
71.2589
71.2143
70.9104
54.5501 CD2Cl2
54.1890 CD2Cl2
53.8280 CD2Cl2
53.4694 CD2Cl2
53.1071 CD2Cl2
38.1311
37.9223
Fe
Fe
f1 (ppm)

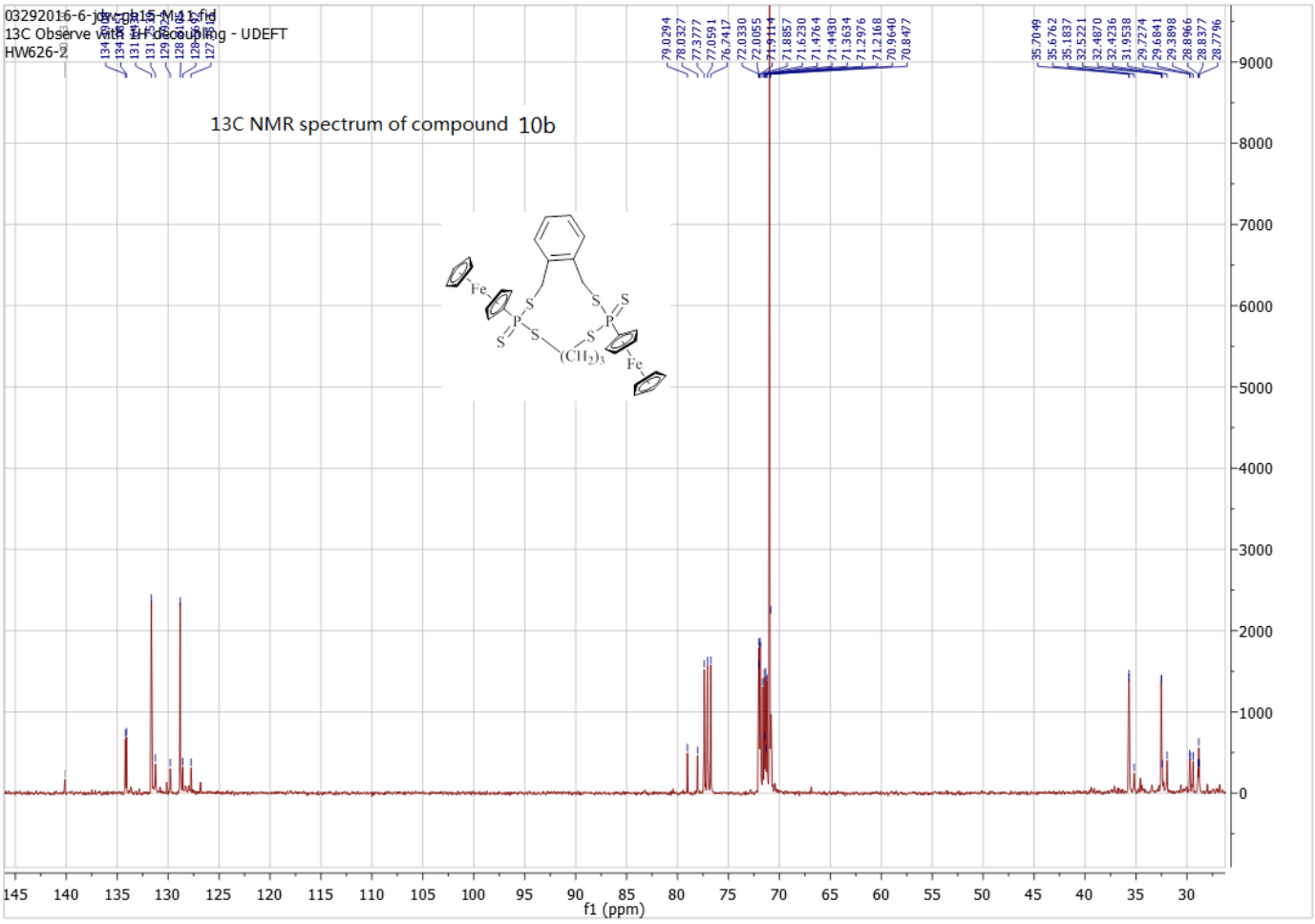

10222013-8-huaI.10.fid
HW339
1H NMR spectrum of compound 9
HDO
f1 (ppm)

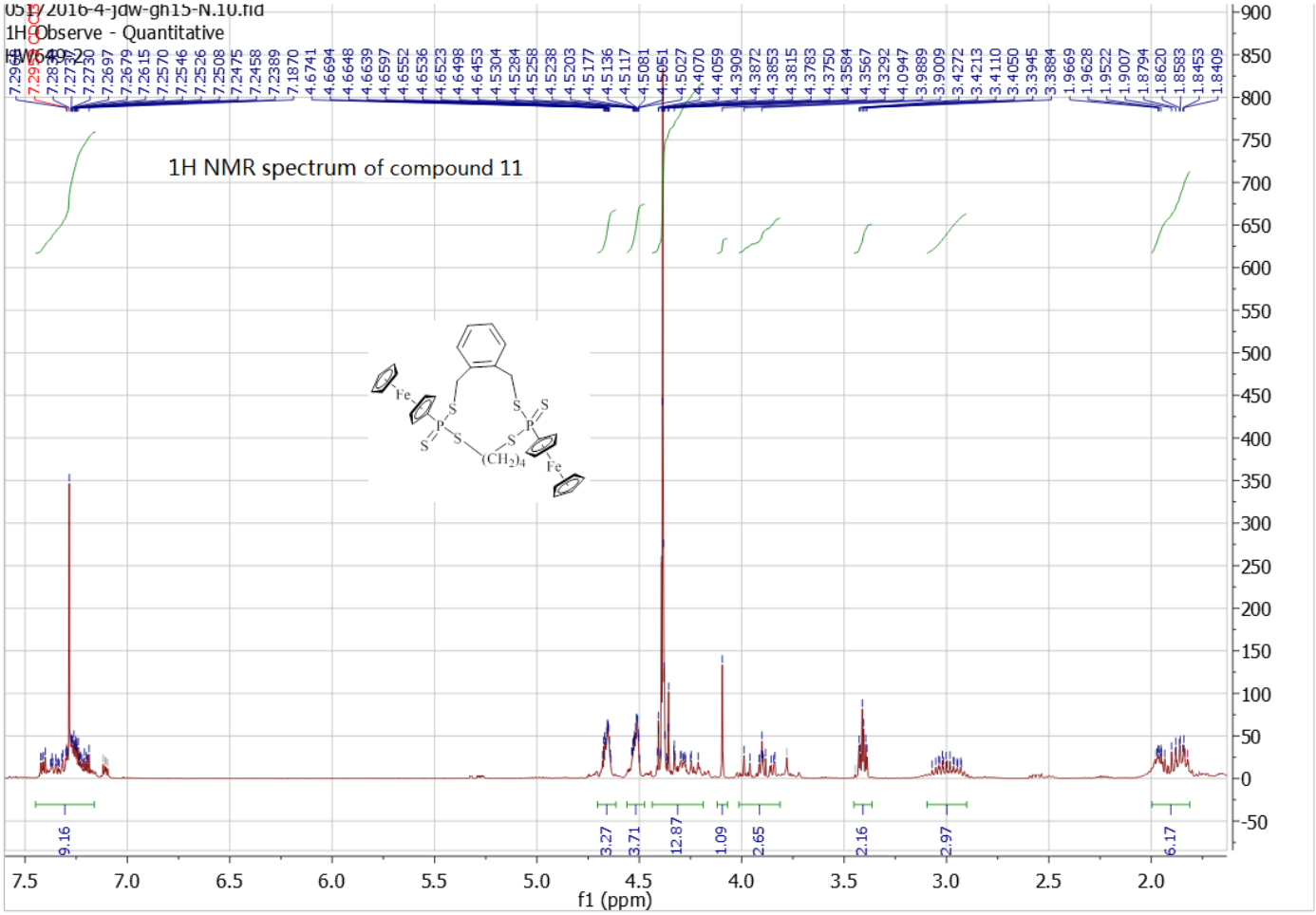

10222013-8-huaI.11.fid
HW339
13C NMR spectrum of compound 9
141.1778
140.1707
139.0428
137.9441
137.6131
135.8629
135.7148
135.0908
134.9538
131.7226
131.4101
131.3550
130.6075
130.2408
128.9831
128.6678
128.4734
126.7450
126.1152
126.0059
82.5960
78.9723
77.6427
73.8246
73.5383
73.3678
72.9269
72.7625
72.7126
72.5595
72.1827
71.9950
71.5110
71.3562
71.3060
71.2105
71.1081
54.5989 CD2Cl2
54.2388 CD2Cl2
53.8784 CD2Cl2
53.5173 CD2Cl2
53.1565 CD2Cl2
36.2855
36.1201
34.9304
31.0217
21.3696
21.2181
f1 (ppm)

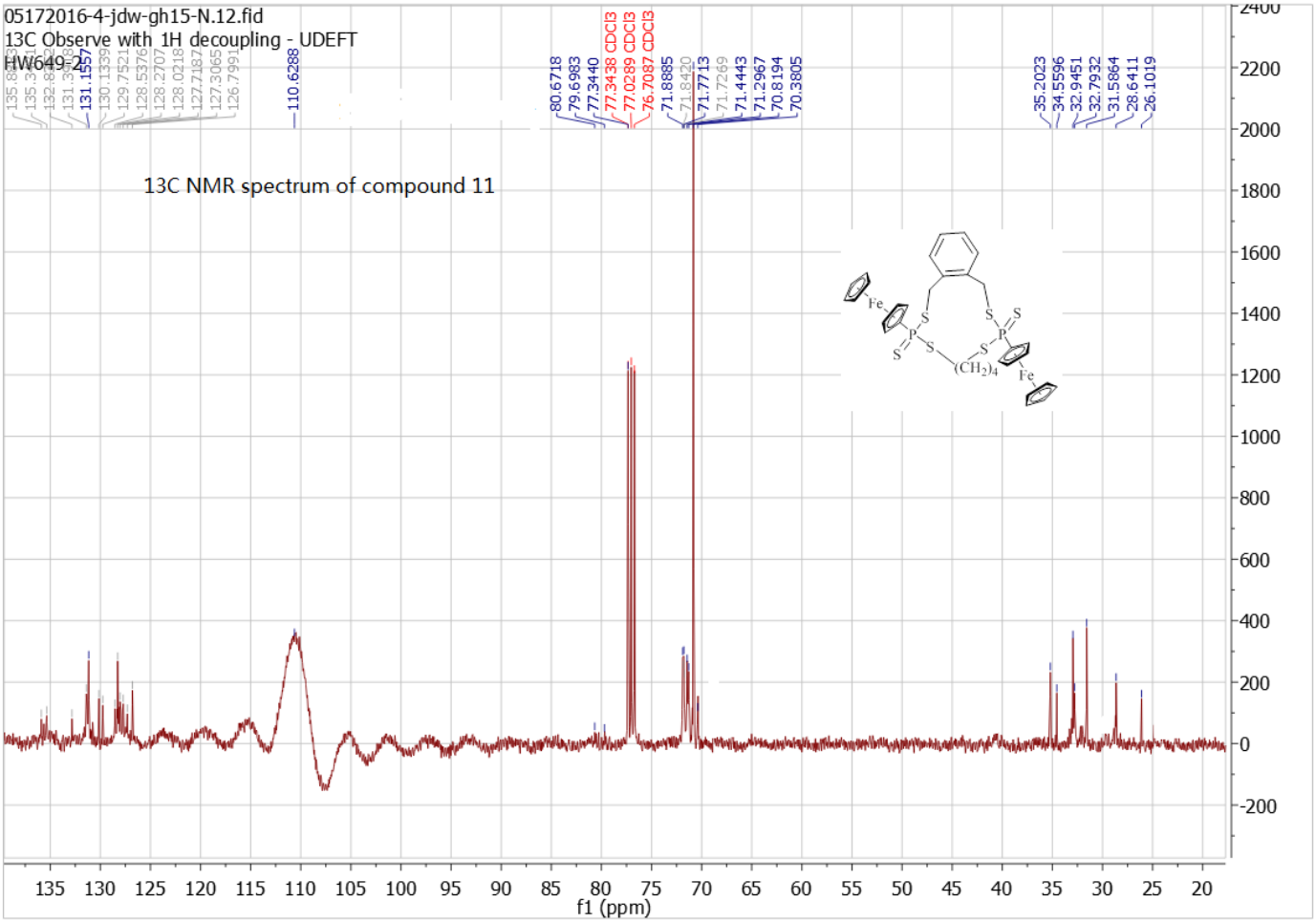

1H NMR spectrum of 10a
f1 (ppm)

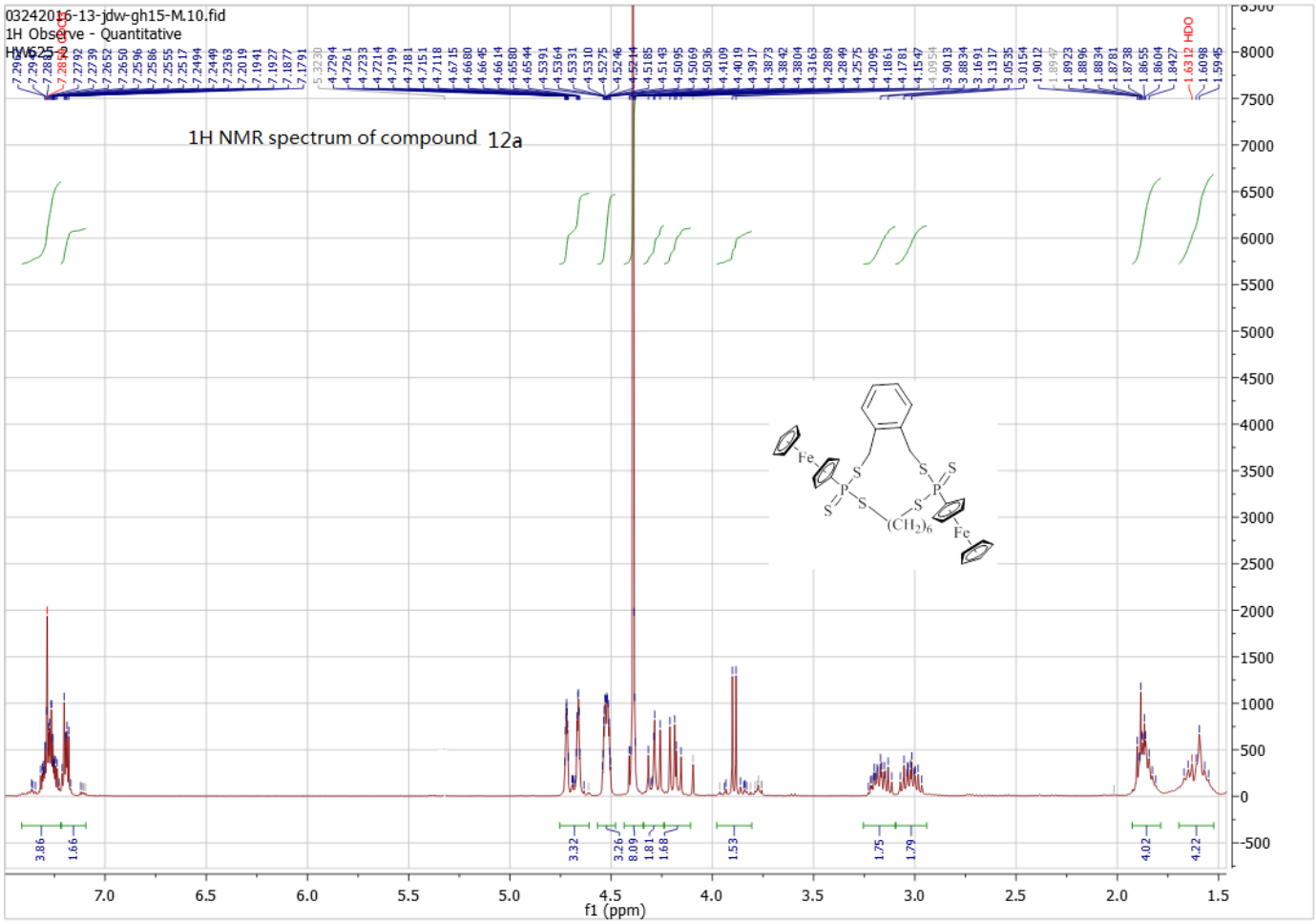

03282016-37-jdw-gh15-M.11.fid
13C Observe with 1H decoupling - UDEFT
HW626-1
13C NMR spectrum of compound 10a
f1 (ppm)

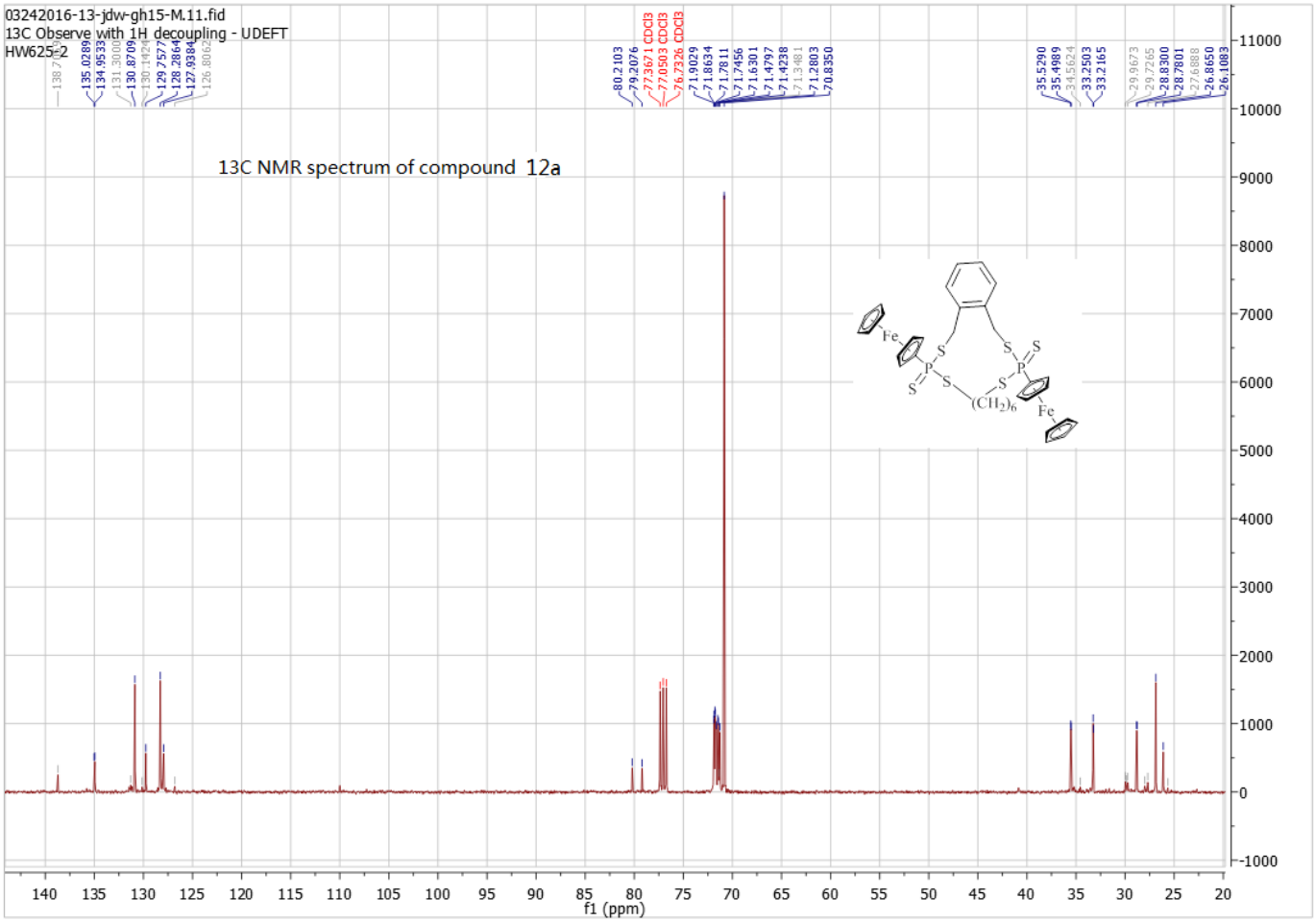

03292016-6-jdw-gh15-M.10.fid
1H Observe - Quantitative
HW626-2
1H NMR spectrum of compound 10b
f1 (ppm)

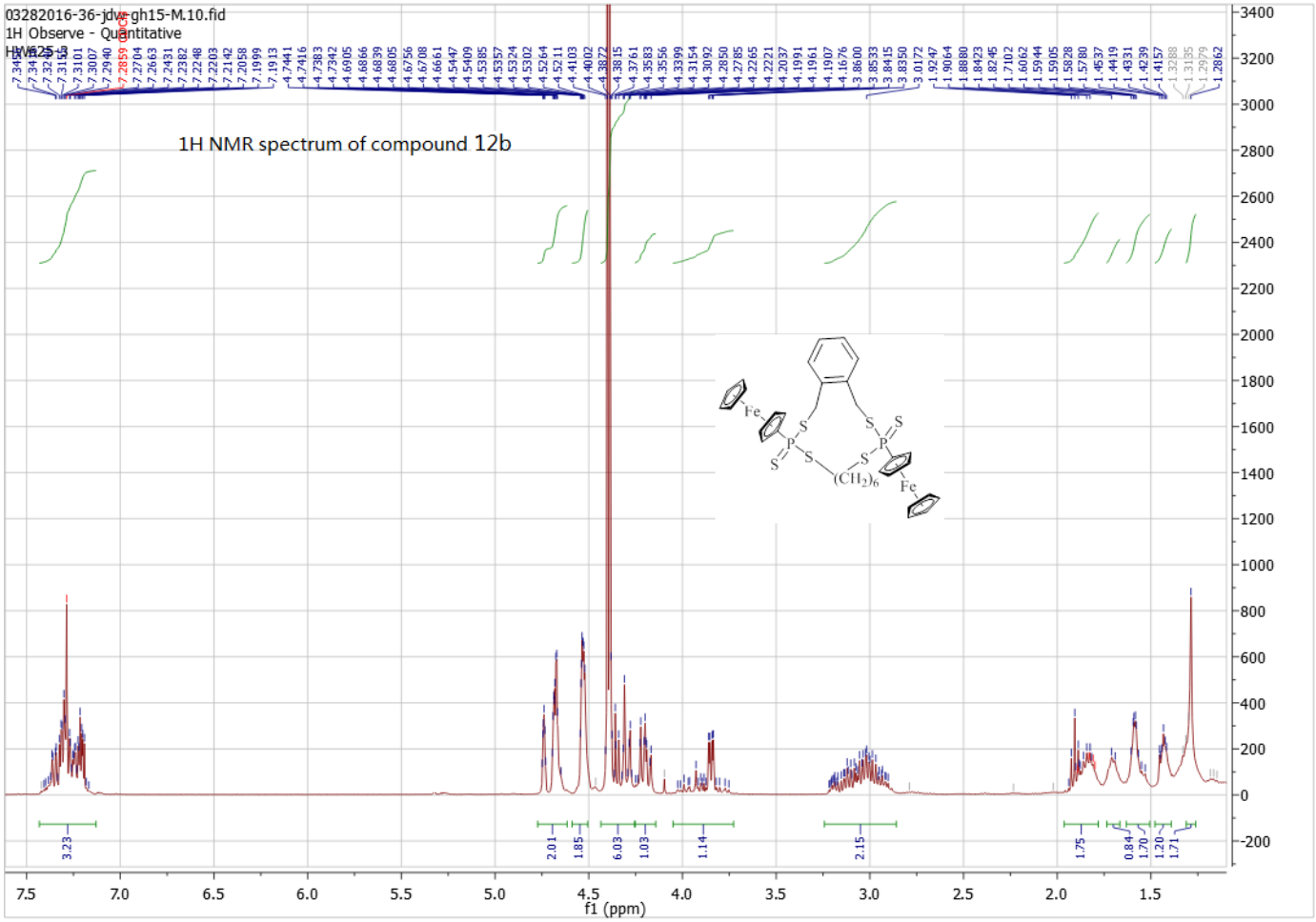

13C NMR spectrum of compound 10b
f1 (ppm)

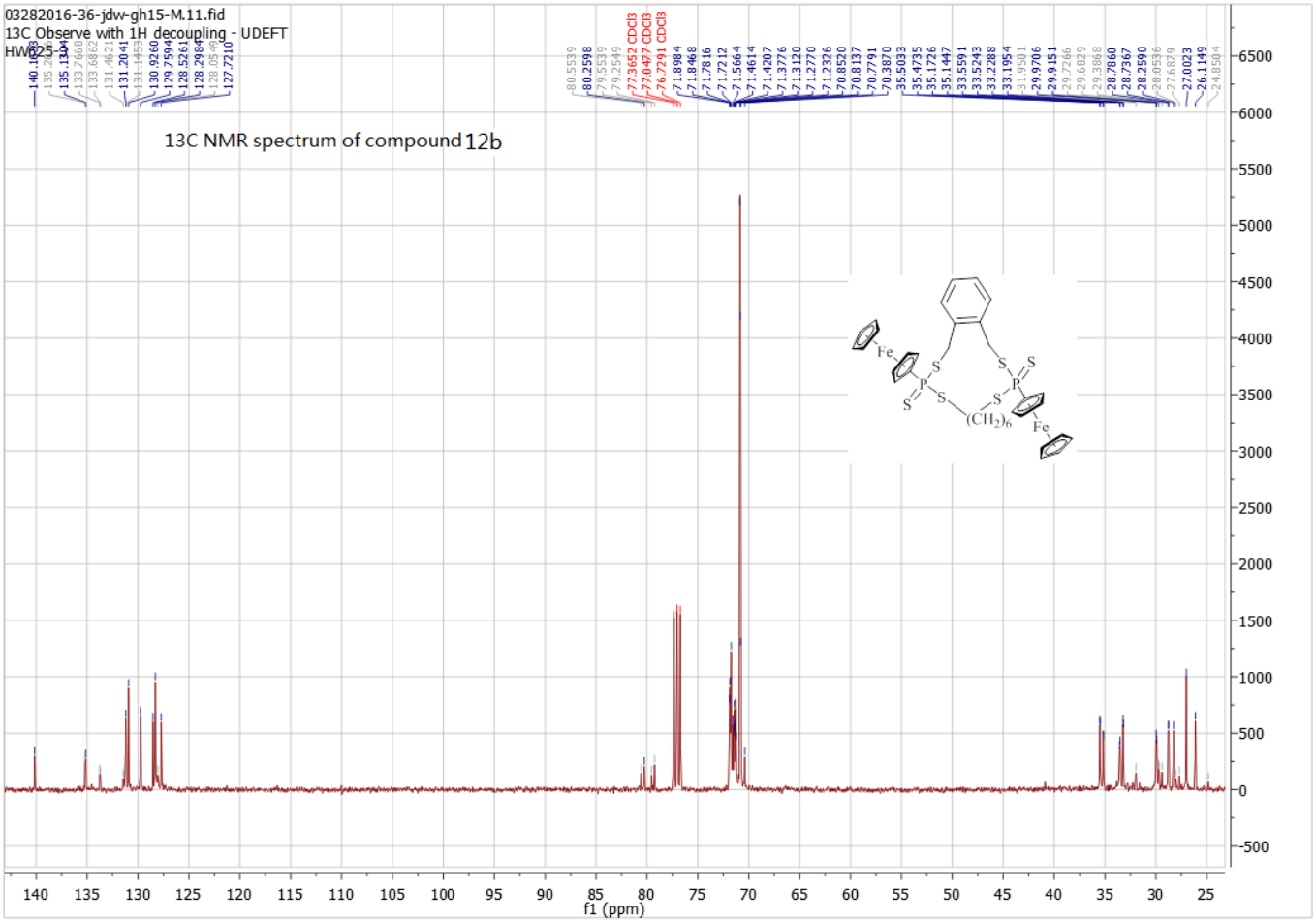

1H NMR spectrum of compound 11
f1 (ppm)

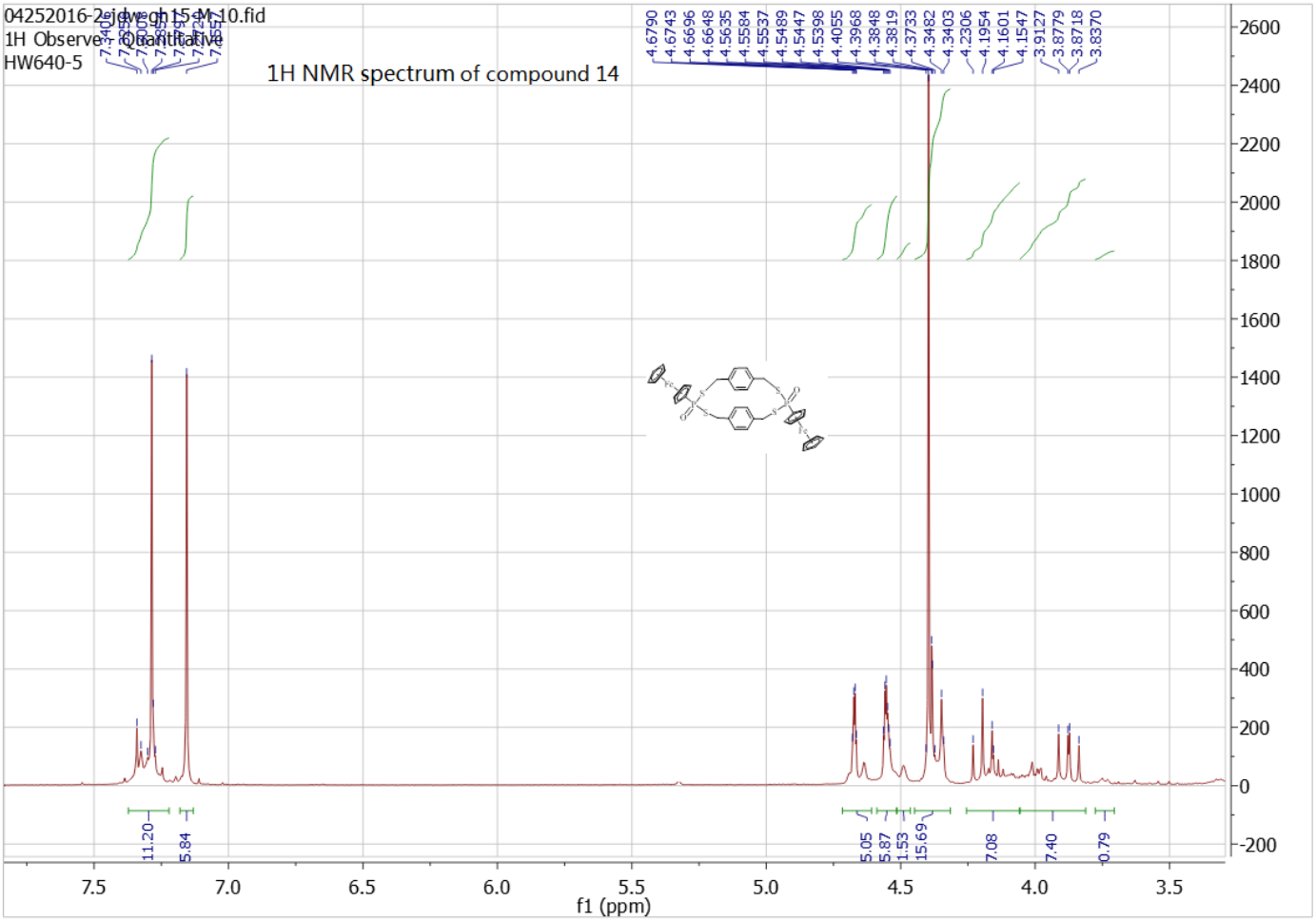

05172016-4-jdw-gh15-N.12.fid
13C Observe with 1H decoupling - UDEFT
HW649-2
13C NMR spectrum of compound 11
f1 (ppm)

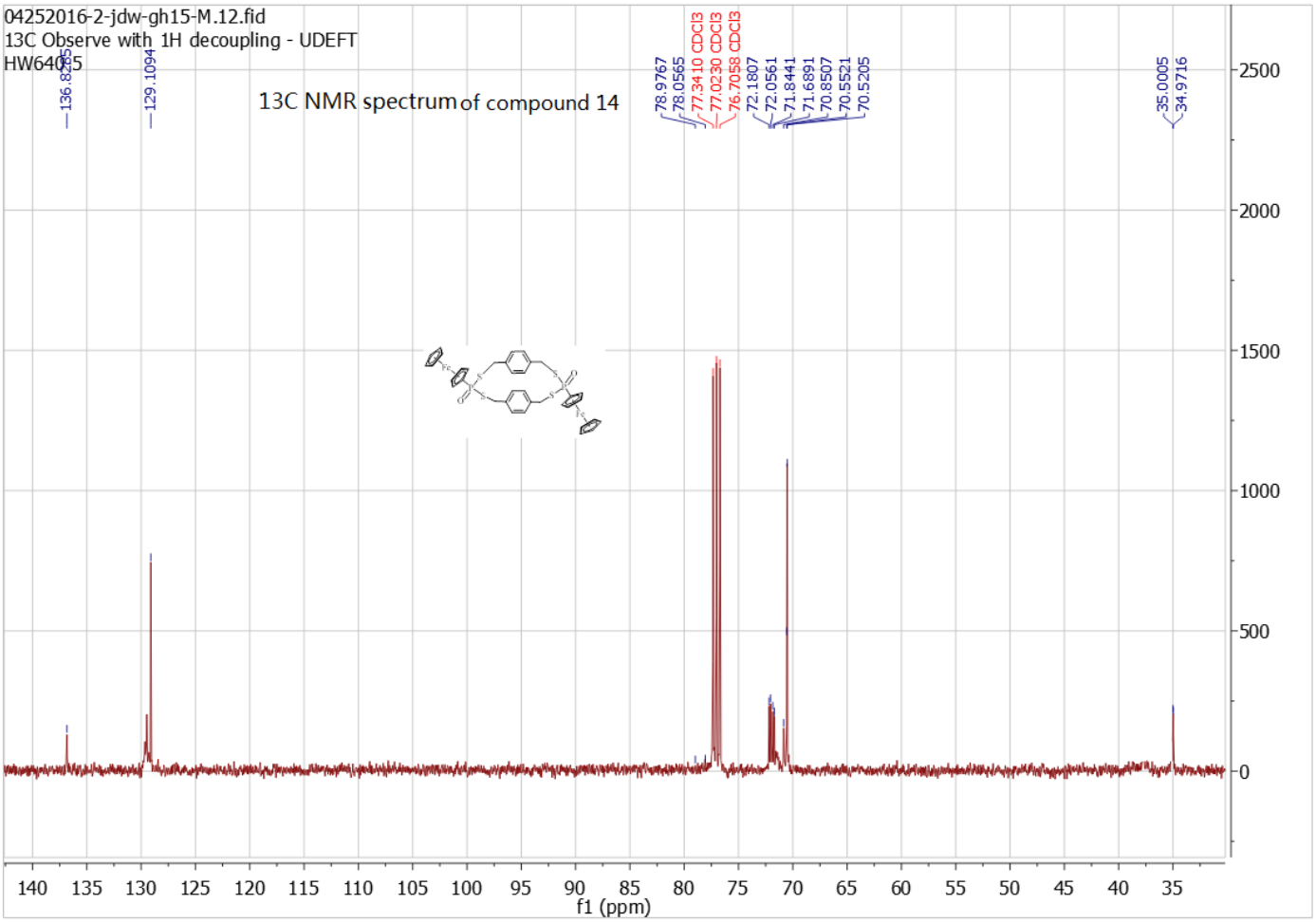

03242016-13-jdw-gh15-M.10.fid
1H Observe - Quantitative
HW625-2
1H NMR spectrum of compound 12a
f1 (ppm)


03242016-13-jdw-gh15-M.11.fid
13C Observe with 1H decoupling - UDEFT
HW625-2
13C NMR spectrum of compound 12a
f1 (ppm)


03282016-36-jdw-gh15-M.10.fid
1H Observe - Quantitative
HW625-3
1H NMR spectrum of compound 12b
f1 (ppm)


03282016-36-jdw-gh15-M.11.fid
13C Observe with 1H decoupling - UDEFT
13C NMR spectrum of compound 12b
f1 (ppm)


04252016-2-jdw-gh15-M-10.fid
1H Observe - Quantitative
HW640-5
1H NMR spectrum of compound 14
f1 (ppm)



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