Supporting Information

One-pot Approach to Organo Phosphorus-Chalcogen Macrocycles Incorporating Double $OP(S)SC_n$ or $OP(Se)SeC_n$ Scaffolds：A Synthetic and Structural Study

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1. ^{1}H, ^{13}C, ^{31}P and ^{77}Se NMR data for compounds 1-17, 19, 20-33 and 40

3,8-Diferrocenyl-1,5,6,10-tetrahydrobenzo[*e*][1,10]dioxa[3,8]dithia[2,9]diphosphacyclododecine 3,8-disulfide (1). ^{1}H NMR (CD_2Cl_2, δ), 7.28-7.23 (m, 4Hx2, Ar-H), 4,86-4.57 (m, 8Hx2, ferrocenyl-H), 4.41 (s, 10H, ferrocenyl-H), 4.42 (s, 10H, ferrocenyl-H), 4.39-4.25 (m, 4Hx2, OCH_2), 4.09-3.96 (m, 4Hx2, SCH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 134.8, 134.7, 134.5, 134.4, 131.2, 131.1, 128.7, 128.6, 76.9 (d, *J*(P,C) = 132.6 Hz), 76.8 (d, *J*(P,C) = 134.9 Hz), 72.2 (d, *J*(P,C) = 14.1 Hz), 71.9 (d, *J*(P,C) = 13.3 Hz), 71.2 (d, *J*(P,C) = 19.3 Hz), 71.0 (d, *J*(P,C) = 18.6 Hz), 70.6, 70.5, 65.0 (d, *J*(P,C) = 14.5 Hz), 64.9 (d, *J*(P,C) = 14.5 Hz), 33.9, 33.8 ppm. ^{31}P NMR (CD_2Cl_2, δ), 95.2 and 95.1 ppm.

4,9-Diferrocenyl-5,8-dioxa-3,10-dithia-4,9-diphospha-1(1,3)-benzenacycloundecaphane 4,9-disulfide (2). ^{1}H NMR (CD_2Cl_2, δ), 7.62 (s, 1H, Ar-H), 7.51 (s, 1H, Ar-H), 7.33-7.24 (m, 3Hx2, Ar-H), 4.86-4.81 (m, 8H, ferrocenyl-H), 4.69-4.58 (m, 8H, ferrocenyl-H), 4.42 (s, 10H, ferrocenyl-H), 4.41 (s, 10H, ferrocenyl-H), 4.17-3.87 (m, 4Hx2, OCH_2), 3.68-3.36 (m, 4Hx2, SCH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 139.4, 139.3, 129.7, 129.6, 128.6, 128.4, 128.3, 128.1, 77.0 (d, *J*(P,C) = 141.8 Hz), 76.5 (d, *J*(P,C) = 138.2 Hz), 72.2 (d, *J*(P,C) = 12.5 Hz), 72.0 (d, *J*(P,C) = 12.1 Hz), 71.3 (d, *J*(P,C) = 14.1 Hz), 71.2 (d, *J*(P,C) = 14.0 Hz), 70.6, 70.5, 63.4, 63.0, 33.7, 33.6 ppm. ^{31}P NMR (CD_2Cl_2, δ), 99.9 and 99.7 ppm.

4,9-Diferrocenyl-5,8-dioxa-3,10-dithia-4,9-diphospha-1(1,4)-benzenacycloundecaphane 4,9-disulfide (3). ^{1}H NMR (CD_2Cl_2, δ), 7.52-7.46 (m, 4H, Ar-H), 7.31-7.20 (m, 4H, Ar-H), 4.59-4.48 (m, 8Hx2, ferrocenyl-H), 4.31 (s, 10H, ferrocenyl-H), 4.30 (s, 10H, ferrocenyl-H), 4.15-3.87 (m, 4Hx2, OCH_2), 3.82-3.65 (m, 4Hx2, SCH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 138.7, 138.0, 129.6, 129.4, 129.3, 76.4 (d, *J*(P,C) = 105.4 Hz), 76.3 (d, *J*(P,C) = 105.6 Hz), 72.3 (d, *J*(P,C) = 14.5 Hz), 72.2 (d, *J*(P,C) = 14.3 Hz), 71.2 (d, *J*(P,C) = 16.1 Hz), 71.1 (d, *J*(P,C) = 16.6 Hz), 70.6, 70.5, 64.4 (d, *J*(P,C) = 10.5 Hz), 64.2(d, *J*(P,C) = 10.9 Hz), 37.7 (d, *J*(P,C) = 7.5 Hz), 37.6 (d, *J*(P,C) = 6.1 Hz)ppm. ^{31}P NMR (CD_2Cl_2, δ), 99.7 and 97.8 ppm.

4,10-Diferrocenyl-5,9-dioxa-3,11-dithia-4,10-diphospha-1(1,3)-benzenacyclododecaphane 4,10-disulfide (4). ^{1}H NMR (CD_2Cl_2, δ), 7.40-7.26 (m, 4Hx2, Ar-H), 4.80-4.53 (m, 8Hx2, ferrocenyl-H), 4.35 (s, 10H, ferrocenyl-H), 4.34 (s, 10H, ferrocenyl-H), 4.32-4.30 (m, 4Hx2, SCH_2), 4.22-3.78 (m, 4Hx2, OCH_2), 1.59-1.52 (m, 2Hx2, CH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 139.7, 129.7, 129.5, 129.1, 128.5, 128.4, 128.1, 77.9 (d, *J*(P,C) = 102.8 Hz), 77.8 (d, *J*(P,C) = 103.0 Hz), 72.1 (d, *J*(P,C) = 10.4 Hz), 72.0 (d, *J*(P,C) = 10.4 Hz), 71.3 (d, *J*(P,C) = 12.8 Hz), 71.2 (d, *J*(P,C) = 14.0 Hz), 70.6, 70.5, 62.1 (d, *J*(P,C) = 7.3 Hz), 61.2 (d, *J*(P,C) = 7.3 Hz), 37.5 (d, *J*(P,C) = 8.3 Hz), 37.4 (d, *J*(P,C) = 8.3 Hz), 30.6 (d, *J*(P,C) = 6.3 Hz), 30.2 (d, *J*(P,C) = 6.3 Hz) ppm. ^{31}P NMR (CD_2Cl_2, δ), 99.0 and 98.9 ppm.

3,9-Diferrocenyl-1,6,7,11-tetrahydro-5H-benzo[*e*][1,10]dioxa[3,8]dithia[2,9]diphosphacyclotridecine 3,9-disulfide (5). ^{1}H NMR (CD_2Cl_2, δ), 7.24-7.03 (m, 4Hx2, Ar-H), 4.70-4.53 (m, 12Hx2, OCH_2+ferrocenyl-H), 4.38 (s, 10H, ferrocenyl-H), 4.32 (s, 10H, ferrocenyl-H), 4.16-4.10 (m, 4H, SCH_2), 3.96-3.82 (m, 4H, SCH_2), 1.53-1.26 (m, 2Hx2, CH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 134.5, 134.3, 131.3, 131.1, 128.8, 128.7, 128.5, 128.3, 76.9 (d, *J*(P,C) = 58.1 Hz), 74.9 (d, *J*(P,C) = 51.9 Hz), 71.9 (d, *J*(P,C) = 11.4 Hz), 71.7 (d, *J*(P,C) = 11.9 Hz), 71.4 (d, *J*(P,C) = 15.5 Hz), 71.3 (d, *J*(P,C) = 15.6 Hz), 70.8, 70.5, 62.4 (d, *J*(P,C) = 8.3 Hz), 60.3 (d, *J*(P,C) = 5.2 Hz), 34.8, 33.6, 31.0, 30.9 ppm. ^{31}P NMR (CD_2Cl_2, δ), 94.2 and 92.4 ppm.

2,9-Diferrocenyl-1,10-dioxa-3,8-dithia-2,9-diphosphacyclotetradecane 2,9-disulfide (6). ^{1}H NMR (CD_2Cl_2, δ), 4.58 (d, *J*(P,H) = 10.7 Hz, 4H, ferrocenyl-H), 4.57 (d, *J*(P,H) = 10.7 Hz, 4H, ferrocenyl-H), 4.44 (d, *J*(P,H) = 3.8 Hz, 4H, ferrocenyl-H), 4.43 (d, *J*(P,H) = 3.8 Hz, 4H, ferrocenyl-H), 4.33 (s, 10H, ferrocenyl-H), 4.32 (s, 10H, ferrocenyl-H), 4.26-4.12 (m, 4Hx2, OCH_2), 3.32-2.65 (m, 4Hx2, SCH_2), 2.02-1.70 (m, 8Hx2, CH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 78.6 (d, *J*(P,C) = 33.2 Hz), 76.5 (d, *J*(P,C) = 30.1 Hz), 72.0 (d, *J*(P,C) = 8.3 Hz), 71.8 (d, *J*(P,C) = 6.2 Hz), 71.5 (d, *J*(P,C) = 12.5 Hz), 71.1 (d, *J*(P,C) = 3.1 Hz), 70.6, 70.5, 63.9 (d, *J*(P,C) = 7.3 Hz), 63.6 (d, *J*(P,C) = 7.3 Hz), 33.5 (d, *J*(P,C) = 3.1 Hz), 32.9 (d, *J*(P,C) = 3.1 Hz), 28.9 (d, *J*(P,C) = 3.1 Hz), 28.5 (d, *J*(P,C) = 3.1 Hz) ppm. ^{31}P NMR (CD_2Cl_2, δ), 99.6, and 97.4 ppm.

3,10-Diferrocenyl-1,5,6,7,8,12-hexahydrobenzo[*e*][1,10]dioxa[3,8]dithia[2,9]diphosphacyclotetradecine 3,10-disulfide (7). ^{1}H NMR (CD_2Cl_2, δ), 7.39-7.19 (m, 4Hx2, Ar-H), 4.68-4.44 (m, 8Hx2, ferrocenyl-H), 4.40 (s, 10H, ferrocenyl-H), 4.38 (s, 10H, ferrocenyl-H), 4.27-4.10 (m, 4Hx2, OCH_2), 4.01-3.90 (m, 4Hx2, SCH_2), 1.90-1.86 (m, 4Hx2, CH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 130.9, 130.8, 128.6, 128.5, 128.1, 128.0, 72.0, 71.8, 71.6, 71.4, 70.5, 70.4, 64.6, 64.5, 35.4, 35.2, 26.7, 26.3 ppm. ^{31}P NMR (CD_2Cl_2, δ), 97.9 and 95.4 ppm.

4,11-Diferrocenyl-5,10-dioxa-3,12-dithia-4,11-diphospha-1(1,3)-benzenacyclotridecaphane 4,11-disulfide (8). ^{1}H NMR (CD_2Cl_2, δ), 7.36-7.24 (m, 4Hx2, Ar-H), 4.74-4.46 (m, 8Hx2, ferrocenyl-H), 4.34 (s,

10H, ferrocenyl-H), 4.32 (s, 10H, ferrocenyl-H), 4.23-3.70 (m, 8Hx2, SCH_2+OCH_2), 1.86-1.41 (m, 4Hx2, CH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 139.8, 139.5, 129.9, 129.8, 129.0, 128.7, 128.2, 127.9, 77.7 (d, J(P,C) = 61.4 Hz), 75.7 (d, J(P,C) = 73.2 Hz), 72.2 (d, J(P,C) = 9.5 Hz), 72.0(d, J(P,C) = 10.3 Hz), 71.6 (d, J(P,C) = 13.6 Hz), 71.4 (d, J(P,C) = 14.1 Hz), 70.6, 70.5, 64.1 (d, J(P,C) = 6.2 Hz), 63.7 (d, J(P,C) = 6.2 Hz), 38.0, 37.9, 26.6 (t, J(P,C) = 3.1 Hz), 25.6 (t, J(P,C) = 3.1 Hz) ppm. ^{31}P NMR (CD_2Cl_2, δ), 98.9, and 98.2 ppm.

4,11-Diferrocenyl-5,10-dioxa-3,12-dithia-4,11-diphospha-1(1,4)-benzenacyclotridecaphane 4,11-disulfide (9). ^1H NMR (CD_2Cl_2, δ), 7.36 (d, J(H,H) = 8.0 Hz, 4H, Ar-H), 7.24 (d, J(H,H) = 8.0 Hz, 4H, Ar-H), 4.57-4.50 (m, 8Hx2, ferrocenyl-H), 4.32 (s, 10H, ferrocenyl-H), 4.31 (s, 10H, ferrocenyl-H), 4.16-3.77 (m, 4Hx2, OCH_2), 3.34 (d, J(H,H) = 8.0 Hz, 4H, SCH_2), 3.12 (d, J(H,H) = 8.0 Hz, 4H, SCH_2), 1.79-1.40 (m, 4Hx2, CH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 138.9, 138.4, 129.4, 129.1, 77.0 (d, J(P,C) = 143.2 Hz), 76.8 (d, J(P,C) = 143.0 Hz), 72.2 (d, J(P,C) = 14.2 Hz), 72.0 (d, J(P,C) = 13.9 Hz), 71.5 (d, J(P,C) = 14.1 Hz), 71.2 (d, J(P,C) = 14.0 Hz), 70.5, 70.4, 64.1 (d, J(P,C) = 7.3 Hz), 63.6 (d, J(P,C) = 6.2 Hz), 37.6, 37.5, 25.9, 25.4 ppm. ^{31}P NMR (CD_2Cl_2, δ), 98.3 and 98.2 ppm.

3,8-Dimethyl-5,6-diphenyl-1,5,6,10-tetrahydrobenzo[*e*][1,10]dioxa[3,8]dithia[2,9]-diphosphacyclododecine 3,8-disulfide (10). ^1H NMR (CD_2Cl_2, δ), 7.35-7.13 (m, 14Hx2, Ar-H), 5.82-5.76 (m, 2Hx2, OCH), 4.53-4.29 (m, 8Hx2, ferrocenyl-H), 4.12 (s, 10H, ferrocenyl-H), 4.11 (s, 10H, ferrocenyl-H), 3.53-3.47 (m, 4Hx2, SCH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 139.2, 136.4, 135.2, 130.9, 130.7, 130.6, 128.4, 128.3, 128.1, 128.0, 127.8, 127.4, 127.3, 127.1, 81.6 (d, J(P,C) = 77.9 Hz), 81.5 (d, J(P,C) = 76.8 Hz), 77.4 (d, J(P,C) = 6.2 Hz), 77.1 (d, J(P,C) = 6.2 Hz), 71.9 (d, J(P,C) = 12.0 Hz), 71.8 (d, J(P,C) = 11.0 Hz), 71.6 (d, J(P,C) = 12.5 Hz), 71.2 (d, J(P,C) = 11.4 Hz), 70.6, 70.4, 35.0 (d, J(P,C) = 23.9 Hz), 31.8 (d, J(P,C) = 22.8 Hz) ppm. ^{31}P NMR (CD_2Cl_2, δ), 100.1 and 100.0 ppm. Mass spectrum (CI^+, m/z), 877 $[M+H]^+$.

4,9-Diferrocenyl-6,7-diphenyl-5,8-dioxa-3,10-dithia-4,9-diphospha-1(1,3)-benzenacycloundecaphane 4,9-disulfide (11). ^1H NMR (CD_2Cl_2, δ), 8.11 (s, 1H, Ar-H), 8.07 (s, 1H, Ar-H), 7.49-7.02 (m, 12Hx2, Ar-H), 6.46-6.34 (m, 2Hx2, OCH), 4.84-4.30 (m, 8Hx2, ferrocenyl-H), 4.23 (s, 10H, ferrocenyl-H), 4.13 (s, 10H, ferrocenyl-H), 3.59-3.30 (m, 4Hx2, SCH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 139.3, 139.2, 136.6, 136.4, 129.7, 129.5, 129.3, 129.1, 128.9, 128.7, 128.5, 128.1, 127.0, 127.4, 127.3, 127.3, 81.5 (d, J(P,C) = 61.4 Hz), 81.4 (d, J(P,C) = 61.0 Hz), 77.3 (d, J(P,C) = 22.6 Hz), 77.2 (d, J(P,C) = 22.0 Hz), 72.1 (d, J(P,C) = 14.5 Hz), 71.9 (d, J(P,C) = 15.5 Hz), 71.5 (d, J(P,C) = 13.5 Hz), 71.0 (d, J(P,C) = 13.0 Hz), 70.5, 70.4, 37.4, 33.2 ppm. ^{31}P NMR (CD_2Cl_2, δ), 100.1 and 99.7 ppm.

6,6-Dibutyl-3,9-diferrocenyl-1,6,7,11-tetrahydro-5*H*-benzo[*e*][1,10]dioxa[3,8]dithia[2,9]-diphosphacyclotridecine 3,9-disulfide (12). ^{1}H NMR (CD_2Cl_2, δ), 7.04 (d, J(H,H) = 7.9 Hz, 2H, Ar-H), 7.03 (d, J(H,H) = 7.9 Hz, 2H, Ar-H), 6.90 (d, J(H,H) = 7.9 Hz, 2H, Ar-H), 6.89 (d, J(H,H) = 7.9 Hz, 2H, Ar-H), 4.60-4.56 (m, 8H, ferrocenyl-H), 4.52-4.43 (m, 8H, ferrocenyl-H), 4.32 (s, 10H, ferrocenyl-H), 4.28 (s, 10H, ferrocenyl-H), 4.17-4.11 (m, 4H, OCH_2), 4.05-3.96 (m, 4H, OCH_2), 3.90-3.82 (m, 4H, SCH_2), 3.79-3.75 (m, 4H, SCH_2), 1.45-1.18 (m, 12Hx2, CH_2), 0.92-0.75 (m, 6Hx2, CH_3) ppm. ^{13}C NMR (CD_2Cl_2, δ), 134.6 (d, J(P,C) = 9.1 Hz), 134.1 (d, J(P,C) = 9.1 Hz), 131.0, 130.9, 128.6, 128.5, 77.2 (d, J(P,C) = 124.2 Hz), 75.8 (d, J(P,C) = 118.0 Hz), 72.4 (d, J(P,C) = 14.6 Hz), 72.0 (d, J(P,C) = 10.5 Hz), 71.5 (d, J(P,C) = 11.7 Hz), 70.8 (d, J(P,C) = 6.5 Hz), 70.6, 70.3, 67.6 (d, J(P,C) = 8.4 Hz), 65.8 (d, J(P,C) = 6.6 Hz), 41.0 (d, J(P,C) = 10.0 Hz), 40.8 (d, J(P,C) = 9.0 Hz), 34.9 (d, J(P,C) = 3.6 Hz), 34.1 (d, J(P,C) = 3.0 Hz), 30.1, 30.0, 24.7, 24.6, 23.7, 23.3, 14.0, 13.9 ppm. ^{31}P NMR (CD_2Cl_2, δ), 94.1 and 93.7 ppm.

7,7-Dibutyl-4,10-diferrocenyl-5,9-dioxa-3,11-dithia-4,10-diphospha-1(1,3)-benzenacyclododecaphane 4,10-disulfide (13). ^{1}H NMR (CD_2Cl_2, δ), 7.31-7.15 (m, 4Hx2, Ar-H), 4.67-4.61 (m, 8H, ferrocenyl-H), 4.52-4.45 (m, 8H, ferrocenyl-H), 4.29 (s, 10H, ferrocenyl-H), 4.27 (s, 10H, ferrocenyl-H), 4.10-3.99 (m, 4H, OCH_2), 3.86-3.71 (m, 4H, OCH_2), 3.65-3.58 (m, 4H, SCH_2), 3.40-3.35 (m, 4H, SCH_2), 1.45-1.10 (m, 12Hx2, CH_2), 0.89-0.80 (m, 6Hx2, CH_3) ppm. ^{13}C NMR (CD_2Cl_2, δ), 139.8, 139.7, 129.9, 129.6, 129.4, 128.4, 128.3, 128.2, 77.8 (d, J(P,C) = 63.9 Hz), 76.4 (d, J(P,C) = 66.8 Hz), 72.3 (d, J(P,C) = 14.0 Hz), 71.9 (d, J(P,C) = 13.4 Hz), 71.4 (d, J(P,C) = 12.4 Hz), 70.9 (d, J(P,C) = 9.0 Hz), 70.4, 70.3, 68.1 (d, J(P,C) = 7.5 Hz), 67.6 (d, J(P,C) = 6.9 Hz), 40.0 (d, J(P,C) = 9.9 Hz), 39.8 (d, J(P,C) = 9.6 Hz), 37.6 (d, J(P,C) = 4.5 Hz), 37.5 (d, J(P,C) = 3.9 Hz), 31.8, 30.6, 30.2, 25.2, 24.7, 23.5, 14.0, 13.9 ppm. ^{31}P NMR (CD_2Cl_2, δ), 98.7 and 98.6 ppm.

7,7-Dibutyl-4,10-diferrocenyl-5,9-dioxa-3,11-dithia-4,10-diphospha-1(1,4)-benzenacyclododecaphane 4,10-disulfide (14). ^{1}H NMR (CD_2Cl_2, δ), 7.39 (d, J(P,H) = 2.0 Hz, 4H, Ar-H), 7.14 (d, J(P,H) = 2.0 Hz, 4H, Ar-H), 4.54-4.52 (m, 8H, ferrocenyl-H), 4.48-4.46 (m, 8H, ferrocenyl-H), 4.45-4.43 (m, 4H, OCH_2), 4.42-4.40 (m, 4H, OCH_2), 4.28 (s, 10H, ferrocenyl-H), 4.26 (s, 10H, ferrocenyl-H), 4.18-4.10 (m, 4H, SCH_2), 3.84-3.74 (m, 4H, SCH_2), 1.46-1.17 (m, 12Hx2, CH_2), 0.89-0.76 (m, 6Hx2, CH_3) ppm. ^{13}C NMR (CD_2Cl_2, δ), 138.7, 138.3, 129.9, 129.8, 129.4, 129.3, 76.8 (d, J(P,C) = 139.5 Hz), 76.7 (d, J(P,C) = 139.5 Hz), 72.1 (d, J(P,C) = 13.6 Hz), 72.0 (d, J(P,C) = 13.1 Hz), 71.1 (d, J(P,C) = 12.0 Hz), 70.9 (d, J(P,C) = 11.7 Hz), 70.4, 70.3, 68.2 (d, J(P,C) = 7.3 Hz), 67.7 (d, J(P,C) = 7.1 Hz), 38.9 (d, J(P,C) = 3.8 Hz), 37.5 (d, J(P,C) = 3.7 Hz), 33.8, 33.5, 25.4, 25.3, 25.1, 23.9, 23.8, 23.6, 14.0, 13.9 ppm. ^{31}P NMR (CD_2Cl_2, δ), 98.6 and 97.4 ppm.

4,11-Diferrocenyl-5,10-dioxa-3,12-dithia-4,11-diphospha-1(1,4)-benzenacyclotridecaphan-7-yne 4,11-disulfide (15). ^{1}H NMR (CD_2Cl_2, δ), 7.39-7.24 (m, 4Hx2, Ar-H), 4.68-4.61 (m, 8Hx2, ferrocenyl-H), 4.57-

4.52 (m, 4Hx2, OCH_2), 4.39 (s, 10H, ferrocenyl-H), 4.38 (s, 10H, ferrocenyl-H), 4.14-4.08 (m, 4Hx2, SCH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 137.7, 137.5, 129.1, 128.3, 81.5 (d, J(P,C) = 11.8 Hz), 81.0 (d, J(P,C) = 12.3 Hz), 76.4 (d, J(P,C) = 138.2 Hz), 76.3 (d, J(P,C) = 140.4 Hz), 72.4 (d, J(P,C) = 14.1 Hz), 72.2 (d, J(P,C) = 14.0 Hz), 72.0 (d, J(P,C) = 12.2 Hz), 71.9 (d, J(P,C) = 12.8 Hz), 70.6, 70.5, 52.9 (d, J(P,C) = 4.1 Hz), 52.7 (d, J(P,C) = 4.1 Hz), 37.5 (d, J(P,C) = 4.2 Hz), 37.1 (d, J(P,C) = 4.3 Hz) ppm. ^{31}P NMR (CD_2Cl_2, δ), 100.5 and 100.4 ppm.

3,8-Diferrocenyl-5,5,6,6-tetraphenyl-1,5,6,10-tetrahydro-benzo[*e*][1,10]dioxa[3,8]dithia[2,9]-diphosphacyclododecine 3,8-disulfide (16). ^{1}H NMR (CD_2Cl_2, δ), 7.79-7.33 (m, 24Hx2, Ar-H), 4.70-4.40 (m, 8Hx2, ferrocenyl-H), 4.24 (s, 10Hx2, ferrocenyl-H), 3.69-3.54 (m, 4Hx2, SCH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 141.5, 141.4, 141.1, 141.0, 137.7, 136.5, 135.6, 135.4, 132.4, 130.9, 130.8, 130.6, 128.8, 128.7, 128.5, 128.3, 128.1, 128.0, 127.8, 127.5, 127.2, 127.1, 79.5, 78.8 (d, J(P,C) = 5.6 Hz), 78.7 (d, J(P,C) = 5.7 Hz), 77.4, 72.0 (d, J(P,C) = 14.0 Hz), 71.9 (d, J(P,C) = 14.2 Hz), 71.4 (d, J(P,C) = 14.0 Hz), 71.3 (d, J(P,C) = 14.1 Hz), 70.4, 70.3, 35.2 (d, J(P,C) = 3.5 Hz), 34.7 (d, J(P,C) = 3.4 Hz) ppm. ^{31}P NMR (CD_2Cl_2, δ), 98.7 and 98.5 ppm.

3-Ferrocenyl-1,5-dihydrobenzo[e][1,3,2]dithiaphosphepine 3-sulfide (17). ^{1}H NMR (CD_2Cl_2, δ), 7.25-7.19 (m, 4H, Ar-H), 4.76 (d, J(P,H) = 14.7 Hz, 1H, SCH_2), 4.72 (d, J(P,H) = 14.7 Hz, 1H, SCH_2), 4.50-4.43 (m, 4H, ferrocenyl-H), 4.30 (s, 5H, ferrocenyl-H), 3.91 (d, J(P,H) = 14.7 Hz, 1H, SCH_2), 3.85 (d, J(P,H) = 14.7 Hz, 1H, SCH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 138.6, 130.0, 128.4, 73.7 (d, J(P,C) = 127 Hz), 72.2 (d, J(P,C) = 12.5 Hz), 70.9 (d, J(P,C) = 15.7 Hz), 70.5, 31.6 (d, J(P,C) = 2.6 Hz) ppm. ^{31}P NMR (CD_2Cl_2, δ), 50.2 ppm.

8,8-Dibutyl-2,5-diferrocenyl-1,6,3,4,2,5-dioxadithiadiphosphonane 2,5-disulfide (19). ^{1}H NMR (CD_2Cl_2, δ), 4.71-4.42 (m, 8H, ferrocenyl-H), 4.24 (s, 10H, ferrocenyl-H), 3.84 (d, J(P,H) = 10.4 Hz, 4H, OCH_2), 1.53-1.17 (m, 12H, CH_2), 0.88 (t, J(H,H) = 7.0 Hz, 6H, CH_3) ppm. ^{13}C NMR (CD_2Cl_2, δ), 72.5, 72.3 (d, J(P,C) = 9.2 Hz), 72.2 (d, J(P,C) = 9.5 Hz), 70.4, 66.4, 56.0, 29.7, 24.7, 23.6, 14.0 ppm. ^{31}P NMR (CD_2Cl_2, δ), 92.6 (s) ppm.

2,7-Bis(4-methoxyphenyl)-1,8,3,6,2,7-dioxadithiadiphosphecane 2,7-disulfide (20). ^{1}H NMR (CD_2Cl_2, δ), 7.90-7.78 (m, 4Hx2, Ar-H), 6.98-6.96 (m, 4Hx2, Ar-H), 4.80-4.16 (m, 4Hx2, OCH_2), 3.83 (s, 6H, OCH_3), 3.82 (s, 6H, OCH_3), 3.49-2.59 (m, 4Hx2, SCH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 163.0, 162.9, 133.4, 133.2, 132.6, 132.4, 132.2, 114.5, 114.3, 114.2, 114.1, 114.0, 113.9, 65.2, 64.5, 55.7, 55.6, 37.7, 33.6 ppm. ^{31}P NMR (CD_2Cl_2, δ), 95.3 and 91.6 ppm.

2,8-Bis(4-methoxyphenyl)-1,9-dioxa-3,7-dithia-2,8-diphosphacycloundecane 2,8-disulfide (21). ^{1}H NMR (CD_2Cl_2, δ), 7.84-7.81 (m, 4Hx3, Ar-H), 6.94-6.92 (m, 4Hx3, Ar-H), 4.70-4.10 (m, 4Hx3, OCH_2), 3.79 (s, 6Hx3, OCH_3), 3.53-2.85 (m, 4Hx3, SCH_2), 2.10-1.92 (m, 2Hx3, CH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 163.1, 133.4, 133.4, 133.1, 132.9, 132.7, 114.3, 114.2, 114.1, 114.0, 65.4, 64.9, 64.2, 55.6, 37.7, 32.7, 31.8, 31.3, 31.0, 30.0 ppm. ^{31}P NMR (CD_2Cl_2, δ), 97.7, 97.5 and 97.0 ppm.

2,9-Bis(4-methoxyphenyl)-1,10-dioxa-3,8-dithia-2,9-diphosphacyclododecane 2,9-disulfide (22). ^{1}H NMR (CD_2Cl_2, δ), 7.88-7.84 (m, 4Hx3, Ar-H), 7.01-6.94 (m, 4Hx3, Ar-H), 4.76-4.25 (m, 4Hx3, OCH_2), 3.83 (s, 6Hx3, OCH_3), 3.63-2.66 (m, 4Hx3, SCH_2), 1.97-0.81 (m, 4Hx3, CH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 163.1, 133.2, 133.1, 133.0, 132.8, 132.7, 114.4, 114.1, 114.0, 113.8, 64.9, 64.8, 64.6, 55.6, 37.7, 33.2, 32.6, 28.7, 28.3, 28.1 ppm. ^{31}P NMR (CD_2Cl_2, δ), 98.1, 97.6 and 96.3 ppm.

3,8-Bis(4-methoxyphenyl)-1,5,6,10-tetrahydrobenzo[*e*][1,10]dioxa[3,8]dithia[2,9]-diphosphacyclododecine 3,8-disulfide (23). ^{1}H NMR (CD_2Cl_2, δ), 7.97-7.89 (m, 4Hx2, Ar-H), 7.18-7.03 (m, 8Hx2, Ar-H), 4.82-4.76 (m, 4Hx2, OCH_2), 4.41-4.30 (m, 4Hx2, SCH_2), 3.87 (s, 6H, OCH_3), 3.86 (s, 6H, OCH_3) ppm. ^{13}C NMR (CD_2Cl_2, δ), 163.4, 134.6, 134.5, 134.3, 134.2, 133.0, 132.8, 132.7, 132.6, 131.3, 131.1, 129.8, 128.9, 128.7, 127.8, 126.5, 126.5, 126.3, 124.6, 124.4, 114.1, 114.2, 65.2, 64.8, 55.6, 34.5, 34.3 ppm. ^{31}P NMR (CD_2Cl_2, δ), 94.0 (big peak) and 93.1 (small peak) ppm.

2,7-Bis(4-methoxyphenyl)-1,8-dioxa-3,6-dithia-2,7-diphosphacycloundecane 2,7-disulfide (24). ^{1}H NMR (CD_2Cl_2, δ), 7.87-7.81 (m, 4Hx2, Ar-H), 7.00-6.97 (m, 4Hx2, Ar-H), 4.47-4.29 (m, 4Hx2, OCH_2), 3.85 (s, 6H, OCH_3), 3.84 (s, 6H, OCH_3), 3.04-2.93 (m, 4Hx2, SCH_2), 2.24-1.25 (m, 2Hx2, CH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 163.1 133.2, 133.1, 132.8, 132.6, 132.4, 132.2, 114.3, 114.1, 64.8 (d, J(P,C) = 8.3 Hz), 61.1 (d, J(P,C) = 6.2 Hz), 64.7, 55.6, 34.0, 33.2, 29.8, 29.7 ppm. ^{31}P NMR (CD_2Cl_2, δ), 93.8 and 92.0 ppm.

4,10-Bis(4-methoxyphenyl)-5,9-dioxa-3,11-dithia-4,10-diphospha-1(1,3)-benzenacyclododecaphane 4,10-disulfide (**25)**. ^{1}H NMR (CD_2Cl_2, δ), 7.96-7.84 (m, 4Hx2, Ar-H), 7.34-7.20 (m, 4Hx2, Ar-H), 7.08-7.00 (m, 4Hx2, Ar-H), 4.21-4.01 (m, 4Hx2, OCH_2), 3.88 (s, 6H, OCH_3), 3.86 (s, 6H, OCH_3), 3.70-3.43 (m, 4Hx2, SCH_2), 1.44-1.25 (m, 2Hx2, CH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 163.2, 139.2, 133.0 (d, J(P,C) = 13.5 Hz), 132.9 (d, J(P,C) = 13.5 Hz), 129.9, 129.6, 129.1, 128.6, 128.4, 128.3, 127.4, 126.5, 126.2, 124.6, 124.3, 114.4, 114.2, 63.0 (d, J(P,C) = 6.2 Hz), 61.3 (d, J(P,C) = 6.2 Hz), 55.6, 37.9 (d, J(P,C) = 4.2 Hz), 37.9 (d, J(P,C) = 4.2 Hz), 30.2, 30.1 ppm. ^{31}P NMR (CD_2Cl_2, δ), 95.5 and 95.4 ppm.

6,6-Dibutyl-3,9-bis(4-methoxyphenyl)-1,6,7,11-tetrahydro-5*H*-benzo[*e*][1,10]dioxa[3,8]dithia[2,9]-diphosphacyclotridecine 3,9-disulfide (26). ^{1}H NMR (CD_2Cl_2, δ), 7.87-7.71 (m, 4Hx2, Ar-H), 7.09-6.86 (m, 8Hx2, Ar-H), 4.18 (d, J(P,H) = 9.9 Hz, 4H, OCH_2), 4.06 (d, J(P,H) = 9.9 Hz, 4H, OCH_2), 3.81 (s, 6H, OCH_3), 3.77 (s, 6H, OCH_3), 3.24 (d, J(P,H) = 11.1 Hz, 4H, SCH_2), 3.23 (d, J(P,H) = 11.1 Hz, 4H, SCH_2), 1.45-1.11 (m, 12Hx2, CH_2), 0.85-0.76 (m, 6Hx2, CH_3) ppm. ^{13}C NMR (CD_2Cl_2, δ), 163.8, 163.7, 135.0 (d, J(P,C) = 56.4 Hz), 134.8 (d, J(P,C) = 56.4 Hz), 133.3 (d, J(P,C) = 13.5 Hz), 133.2 (d, J(P,C) = 13.5 Hz), 131.8, 133.1, 129.6, 129.4, 127.2 (d, J(P,C) = 3.1 Hz), 125.5 (d, J(P,C) = 3.1 Hz), 115.3, 115.1, 115.0, 114.8, 70.1 (d, J(P,C) = 8.5 Hz), 66.7 (d, J(P,C) = 8.5 Hz), 55.4, 55.3, 35.8, 35.1, 31.6, 30.8, 24.8, 24.2, 14.6, 14.5 ppm. ^{31}P NMR (CD_2Cl_2, δ), 90.6 and 88.2 ppm.

7,7-Dibutyl-4,10-bis(4-methoxyphenyl)-5,9-dioxa-3,11-dithia-4,10-diphospha-1(1,3)-benzenacyclododecaphane 4,10-disulfide (27). ^{1}H NMR (CD_2Cl_2, δ), 7.88-7.77 (m, 4Hx2, Ar-H), 7.27-6.93 (m, 8Hx2, Ar-H), 4.12-4.01 (m, 4Hx2, OCH_2), 3.80 (s, 6H, OCH_3), 3.78 (s, 6H, OCH_3), 3.74-3.43 (m, 4Hx2, SCH_2), 1.19-0.77 (m, 12Hx2, CH_2), 0.72 (t, J(H,H) = 7.3 Hz, 6H, CH_3), 0.65 (t, J(H,H) = 7.1 Hz, 6H, CH_3) ppm. ^{13}C NMR (CD_2Cl_2, δ), 163.2, 163.1, 139.5, 139.4, 133.0 (d, J(P,C) = 13.2 Hz), 132.9 (d, J(P,C) = 13.3 Hz), 129.8, 129.5, 128.7, 128.3, 126.7, 126.3, 125.8, 124.5, 114.3 (d, J(P,C) = 3.1 Hz), 114.1 (d, J(P,C) = 3.1 Hz), 70.1 (d, J(P,C) = 8.5 Hz), 66.7 (d, J(P,C) = 8.5 Hz), 55.4, 55.3, 41.6, 40.8, 35.8, 35.1, 29.8, 29.7, 24.8, 24.7, 23.5, 23.4, 14.6, 14.5 ppm. ^{31}P NMR (CD_2Cl_2, δ), 94.9 and 94.8 ppm.

7,7-Dibutyl-4,10-bis(4-methoxyphenyl)-5,9-dioxa-3,11-dithia-4,10-diphospha-1(1,4)-benzenacyclododecaphane 4,10-disulfide (28). ^{1}H NMR (CD_2Cl_2, δ), 7.72 (dd, J(P,H) = 13.2 Hz, J(H,H) = 8.5 Hz, 4H, Ar-H), 7.71 (dd, J(P,H) = 13.2 Hz, J(H,H) = 8.5 Hz, 4H, Ar-H), 7.41 (dd, J(P,H) = 3.1 Hz, J(H,H) = 8.2 Hz, 4H, Ar-H), 7.11 (dd, J(P,H) = 3.1 Hz, J(H,H) = 8.2 Hz, 4H, Ar-H), 6.99-6.92 (m, 4Hx2, Ar-H), 4.21-4.13 (m, 4H, OCH_2), 4.02-3.93 (m, 4H, OCH_2), 3.80 (s, 6H, OCH_3), 3.79 (s, 6H, OCH_3), 3.76-3.45 (m, 4Hx2, SCH_2), 1.47-0.65 (m, 18Hx2, CH_2+CH_3) ppm. ^{13}C NMR (CD_2Cl_2, δ), 163.1, 163.0, 138.6, 138.0, 132.7 (d, J(P,C) = 13.1 Hz), 132.6 (d, J(P,C) = 13.0 Hz), 129.8, 129.9, 129.4, 129.3, 126.2, 126.1, 124.9, 124.8, 114.3, 114.0, 68.5 (d, J(P,C) = 7.3 Hz), 68.0 (d, J(P,C) = 7.2 Hz), 55.6, 55.5, 40.4 (d, J(P,C) = 9.5 Hz), 40.2 (d, J(P,C) = 9.5 Hz), 38.3, 38.2, 33.4, 32.4, 25.0, 24.9, 23.6, 23.5, 13.9, 13.8 ppm. ^{31}P NMR (CD_2Cl_2, δ), 94.0 and 93.6 ppm.

4,11-Bis(4-methoxyphenyl)-5,10-dioxa-3,12-dithia-4,11-diphospha-1(1,4)-benzenacyclotridecaphan-7-yne 4,11-disulfide (29). ^{1}H NMR (CD_2Cl_2, δ), 7.96-7.87 (m, 4Hx2, Ar-H), 7.32-7.30 (m, 4Hx2, Ar-H), 7.11-7.07 (m, 4Hx2, Ar-H), 4.72-4.40 (m, 4H, OCH_2), 4.19-4.02 (m, 4H, OCH_2), 3.91 (s, 6H, OCH_3), 3.90 (s, 6H, OCH_3), 3.71-3.54 (m, 4Hx2, SCH_2) ppm. ^{13}C NMR (CD_2Cl_2, δ), 164.1, 164.0, 138.1, 138.0, 133.6 (d, J(P,C) =

13.5 Hz), 133.5 (d, *J*(P,C) = 13.5 Hz), 129.9, 129.8, 115.2, 115.0, 82.0, 81.8, 58.4, 58.3, 53.8, 53.7, 38.6, 38.5 ppm. ^{31}P NMR (CD_2Cl_2, δ), 96.6 and 96.5 ppm.

6,6-Dibutyl-3,9-diphenyl-1,6,7,11-tetrahydro-5H-benzo[*e*][1,10]dioxa[3,8]diselena[2,9]diphosphacyclotridecine 3,9-diselenide (30). ^{1}H NMR (CD_2Cl_2, δ), 8.08-7.02 (m, 14Hx2, Ar-H), 4.02 (d, *J*(P,H) = 11.5 Hz, 4H, OCH_2), 3.98 (d, *J*(P,H) = 11.5 Hz, 4H, OCH_2), 3.48 (d, *J*(P,H) = 9.9 Hz, 4H, $SeCH_2$), 3.47 (d, *J*(P,H) = 9.9 Hz, 4H, $SeCH_2$), 1.55-1.26 (m, 12Hx2, CH_2), 1.02-0.89 (m, 6Hx2, CH_3) ppm. ^{13}C NMR (CD_2Cl_2, δ), 140.4, 140.3, 136.0 (d, *J*(P,C) = 95.0 Hz), 135.8 (d, *J*(P,C) = 99.1 Hz), 133.0 (d, *J*(P,C) = 3.1 Hz), 129.9 (d, *J*(P,C) = 3.1 Hz), 129.4, 129.1, 126.4, 126.1, 124.9, 124.0, 114.9, 114.8, 68.5, 38.2, 37.9, 30.8, 30.5, 28.8, 27.9, 23.5, 23.2, 14.7, 14.6 ppm. ^{31}P NMR (CD_2Cl_2, δ), 73.8 (s, *J*(P-Se) = 460 Hz, *J*(P=Se) = 821 Hz), 67.3 (s, *J*(P-Se) = 460 Hz, *J*(P=Se) = 821 Hz) ppm. ^{77}Se NMR (CD_2Cl_2, δ), 439.5 (d, ^{1}J(P-Se) = 460 Hz), 439.0 (d, ^{1}J(P-Se) = 460 Hz), -57.4 (d, *J*(P,Se) = 821 Hz), -87.8 (d, *J*(P,Se) = 821 Hz) ppm.

7,7-Dibutyl-4,10-diphenyl-5,9-dioxa-3,11-diselena-4,10-diphospha-1(1,3)-benzenacyclododecaphane 4,10-diselenide (31). ^{1}H NMR (CD_2Cl_2, δ), 8.04-7.90 (m, 4Hx2, Ar-H), 7.56-6.99 (m, 10Hx2, Ar-H), 4.19-3.75 (m, 4Hx2, OCH_2), 3.59-2.88 (m, 4Hx2, $SeCH_2$), 1.41-1.09 (m, 12Hx2, CH_2), 0.96-0.74 (m, 6Hx2, CH_3) ppm. ^{13}C NMR (CD_2Cl_2, δ), 140.8, 140.7, 136.6 (d, *J*(P,C) = 98.8 Hz), 136.4 (d, *J*(P,C) = 100.9 Hz), 133.4 (d, *J*(P,C) = 3.1 Hz), 133.3 (d, *J*(P,C) = 3.1 Hz), 133.2 4, 131.4, 131.3, 131.2, 131.1, 131.0, 130.4, 130.1, 129.6, 129.4, 70.5, 70.4, 69.7, 69.6, 41.2, 41.0, 36.8, 36.6, 25.3, 25.2, 24.1, 24.0, 14.6, 14.5 ppm. ^{31}P NMR (CD_2Cl_2, δ), 79.9 (s, *J*(P-Se) = 450 Hz, *J*(P=Se) = 830 Hz), 79.6 (s, *J*(P-Se) = 450 Hz, *J*(P=Se) = 826 Hz) ppm. ^{77}Se NMR (CD_2Cl_2, δ), 478.3 (d, ^{1}J(P-Se) = 450 Hz), 464.4 (d, ^{1}J(P-Se) = 450 Hz), -100.4 (d, *J*(P,Se) = 830 Hz), -104.8 (d, *J*(P,Se) = 830 Hz) ppm.

7,7-Dibutyl-4,10-diphenyl-5,9-dioxa-3,11-diselena-4,10-diphospha-1(1,4)-benzenacyclododecaphane 4,10-diselenide (32). ^{1}H NMR (CD_2Cl_2, δ), 7.83 (d, *J*(P,H) = 7.9 Hz, 4H, Ar-H), 7.79 (d, *J*(P,H) = 7.9 Hz, 4H, Ar-H), 7.51-7.42 (m, 8Hx2, Ar-H), 6.90-6.85 (m, 2Hx2, Ar-H), 4.12-3.60 (m, 4Hx2, OCH_2), 2.91-2.79 (m, 4Hx2, $SeCH_2$), 1.33-1.01 (m, 12Hx2, CH_2), 0.88-0.69 (m, 6Hx2, CH_3) ppm. ^{13}C NMR (CD_2Cl_2, δ), 139.7, 139.1, 136.0 (d, *J*(P,C) = 100.1 Hz), 135.9 (d, *J*(P,C) = 100.7 Hz), 133.8, 133.4, 133.3 (d, *J*(P,C) = 3.1 Hz), 133.1 (d, *J*(P,C) = 3.1 Hz), 130.8, 130.3, 129.7, 129.6, 129.5, 129.4, 70.3, 70.2, 69.1, 69.0, 41.1, 41.0, 37.0, 36.9, 33.9, 33.4, 25.9, 24.3, 14.6, 14.5 ppm. ^{31}P NMR (CD_2Cl_2, δ), 78.7 (s, *J*(P-Se) = 440 Hz, *J*(P=Se) = 812 Hz), 77.5 (s, *J*(P-Se) = 440 Hz, *J*(P=Se) = 812 Hz) ppm. ^{77}Se NMR (CD_2Cl_2, δ), 480.8 (d, ^{1}J(P-Se) = 440 Hz), 448.5 (d, ^{1}J(P-Se) = 440 Hz), -71.4 (d, *J*(P,Se) = 812 Hz), -94.9 (d, *J*(P,Se) = 812 Hz) ppm.

11,11-Dibutyl-2,8-diphenyl-1,9-dioxa-3,7-diselena-2,8-diphosphacyclododecane 2,8-diselenide (33). ^{1}H NMR (CD_2Cl_2, *δ*), 7.94-7.77 (m, 4Hx2, Ar-H), 7.50-7.43 (m, 6Hx2, Ar-H), 4.13-3.89 (m, 4Hx2, OCH_2), 3.79-3.74 (m, 4Hx2, $SeCH_2$), 3.50-3.45 (m, 2H, CH_2), 3.23-3.19 (m, 2H, CH_2), 1.34-1.18 (m, 12Hx2, CH_2), 0.89-0.75 (m, 6Hx2, CH_3) ppm. ^{13}C NMR (CD_2Cl_2, *δ*), 136.6 (d, *J*(P,C) = 95.8 Hz), 136.2 (d, *J*(P,C) = 97.0 Hz), 133.3 (d, *J*(P,C) = 3.1 Hz), 133.2 (d, *J*(P,C) = 3.1 Hz), 131.1 (d, *J*(P,C) = 12.5 Hz), 131.0 (d, *J*(P,C) = 12.5 Hz), 129.6, 129.4, 71.6, 71.5, 68.0, 67.9, 41.7, 41.6, 33.8, 33.6, 30.5, 29.9, 25.2, 25.0, 24.1, 24.0, 14.7, 14.5 ppm. ^{31}P NMR (CD_2Cl_2, *δ*), 78.7 (s, *J*(P-Se) = 465 Hz, *J*(P=Se) = 825 Hz), 76.8 (s, *J*(P-Se) = 440 Hz, *J*(P=Se) = 825 Hz) ppm. ^{77}Se NMR (CD_2Cl_2, *δ*), 356.7 (d, ^{1}J(P-Se) = 465 Hz), 338.0 (d, ^{1}J(P-Se) = 465 Hz), -79.3 (d, *J*(P,Se) = 825 Hz), -93.2 (d, *J*(P,Se) = 825 Hz) ppm.

4-Phenyl-3.5-diselena-4-phosphabicyclo[5.4.0]undeca-1(7),8,10-triene-4-selone (37). ^{1}H NMR (CD_2Cl_2, *δ*), 8.18-7.24 (m, 9H, ArH), 4.67 (d, *J*(P,H) = 13.5 Hz, 4H, $SeCH_2$) ppm. ^{13}C NMR (CD_2Cl_2, *δ*), 139.0, 133.3 (d, *J*(P,C) = 3.1 Hz), 131.7 (d, *J*(P,C = 11.4 Hz), 130.4, 129.2 (d, *J*(P,C) = 14.5 Hz), 128.4, 32.4 ppm. ^{31}P NMR (CD_2Cl_2, *δ*), 14.0 (s, *J*(P,Se) = 369 Hz, *J*(P,Se) = 770 Hz) ppm. ^{77}Se NMR (CD_2Cl_2, *δ*), 302.0 (d, *J*(P,Se) = 369 Hz), -393.1 (d, *J*(P,Se) = 770 Hz) ppm.

2,4,5-Triphenyl-1,3,2-dioxaphospholane 2-selenide (38). ^{1}H NMR (CD_2Cl_2, δ), 7.92-7.08 (m, 15H, ArH), 5.75 (d, 2H, CH) ppm. ^{13}C NMR (CD_2Cl_2, δ), 134.1, 132.8, 130.7, 130.2, 130.0, 128.6, 128.3, 128.0, 127.2, 85.0 ppm. ^{31}P NMR (CD_2Cl_2, δ), 107.9 (s, *J*(P,Se) = 925 Hz) ppm. ^{77}Se NMR (CD_2Cl_2, δ), -186.0 (d, *J*(P,Se) = 925 Hz) ppm.

8,8-Dibutyl-2,5-diphenyl-1,6,3,4,2,5-dioxadiselenadiphosphonane 2,5-diselenide (40). ^{1}H NMR (CD_2Cl_2, *δ*), 8.00-7.92 (m, 2H, Ar-H), 7.54-7.50 (m, 3H, Ar-H), 7.37-7.24 (m, 3H, Ar-H), 7.20-7.10 (m, 2H, Ar-H), 3.98 (d, *J*(P,H) = 9.9 Hz, 4H, OCH_2), 1.53-1.25 (m, 12H, CH_2), 0.87 (t, *J*(H,H) = 7.0 Hz, 6H, CH_3) ppm. ^{13}C NMR (CD_2Cl_2, *δ*), 135.3 (d, *J*(P,C) = 102.8 Hz), 130.6 (d, *J*(P,C) = 12.5 Hz), 128.7 (d, *J*(P,C) = 3.1 Hz), 126.5, 68.5, 56.1, 30.0, 24.7, 23.4, 13.9 ppm. ^{31}P NMR (CD_2Cl_2, *δ*), 67.3 (s, *J*(P-Se) = 465 Hz, *J*(P=Se) = 812 Hz) ppm. ^{77}Se NMR (CD_2Cl_2, *δ*), 439.5 (d, ^{1}J(P-Se) = 465 Hz, ^{2}J(P-Se) = 21.5 Hz), -57.5 (d, *J*(P,Se) = 812 Hz) ppm.

2. **Details of the X-ray data collections and refinements for 3, 4, 5, 10, 11, 26, 29, 30 and 40**

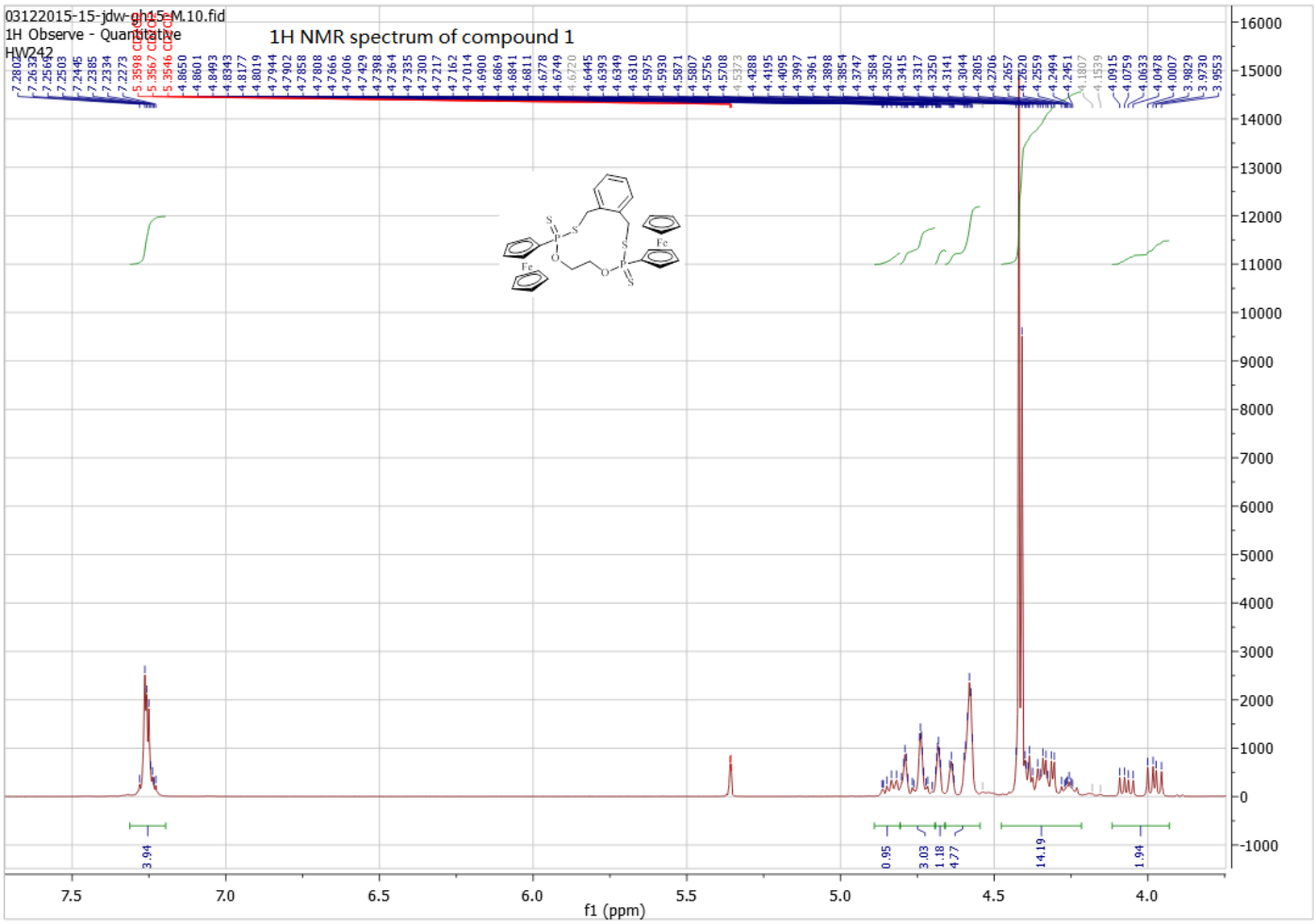
Table S1. Details of the X-ray data collections and refinements for **3**, **4**, **5** and **10**

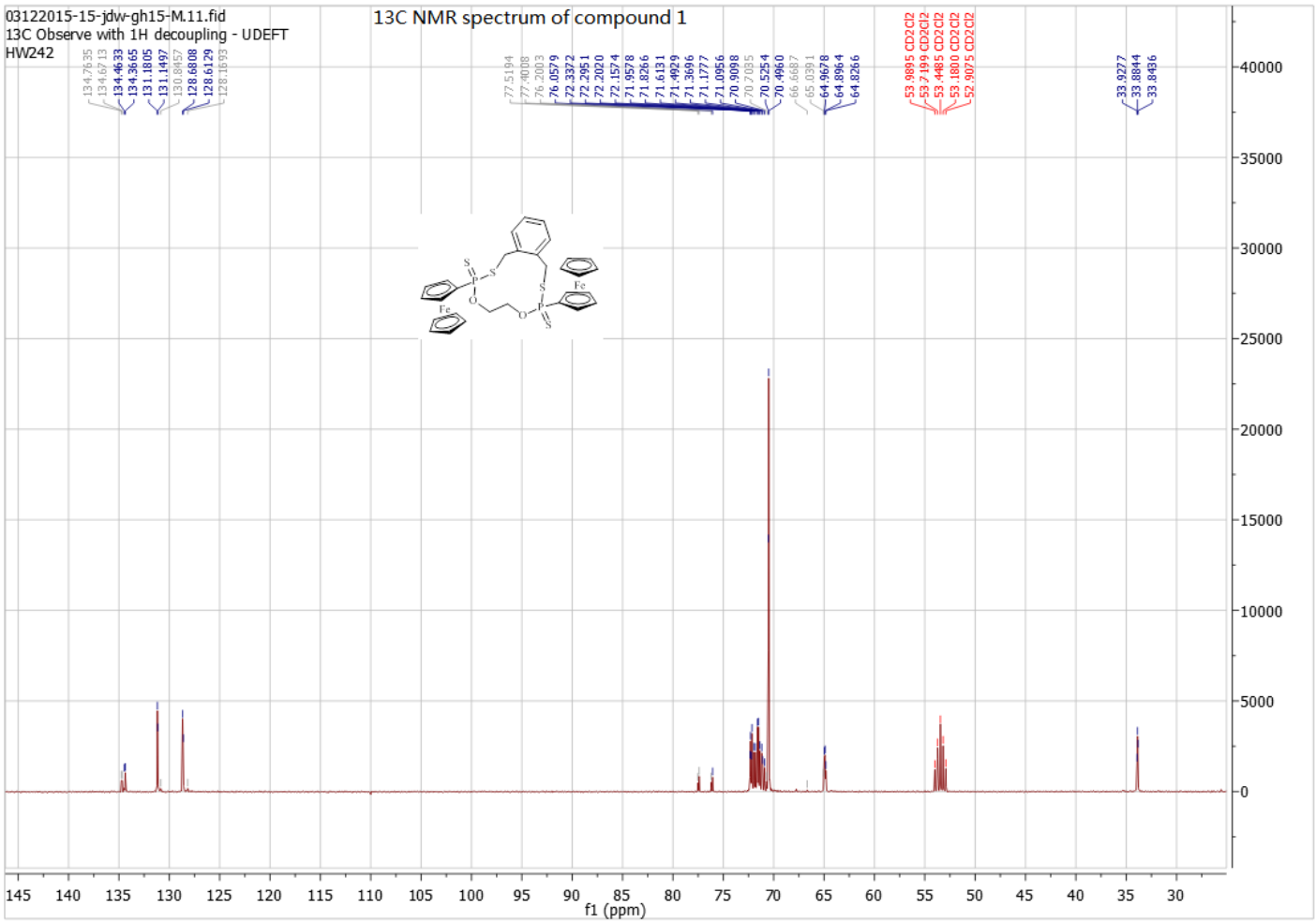
Compound	**3**	**4**	**5**	**10**
Formula	$C_{30}H_{30}Fe_2O_2P_2S_4$	$C_{34}H_{38}Fe_2O_3P_2S_4$	$C_{31}H_{32}Fe_2O_2P_2S_4$	$C_{42}H_{38}Fe_2O_2P_2S_4$
M	724.45	796.55	738.47	876.64
Crystal system	Triclinic	Monoclinic	Monoclinic	Orthorhombic
Space group	*P-1*	$P2_1/c$	$P2_1/c$	*Pbca*
a/Å	10.079(2)	14.260(3)	28.792(9)	12.4114(17)
b/Å	10.892(3)	10.367(2)	7.574(2)	22.626(3)
c/Å	14.675(3)	24.945(6)	14.883(5)	28.013(3)
A	79.83(2)	90	90	90
B	72.080(18)	105.930(6)	94.755(8)	90
Γ	86.68(3)	90	90	90
U/A^3	1508.8(7)	3545.9(14)	3234.5(17)	7866.7(18)
Z	2	4	4	8
μ/mm^{-1}	13.717	11.768	12.813	10.669
Reflections collected	9694	26065	16283	57019
Independent reflections	5134	6212	5544	6894
R_{int}	0.0975	0.1341	0.1390	0.0836
R1	0.0728	0.0757	0.0730	0.0736
wR2 [I > 2σ(I)]	0.2318	0.1457	0.1717	0.1714

Table S2. Details of the X-ray data collections and refinements for **11**, **26**, **29**, **30** and **40**

Compound	**11**	**26**	**29**	**30**	**40**
Formula	$C_{42}H_{38}Fe_2O_2P_2S_4$	$C_{33}H_{44}O_4P_2S_4$	$C_{26}H_{26}O_4P_2S_4$	$C_{31}H_{40}O_2P_2Se_4$	$C_{23}H_{32}O_2P_2Se_4$
M	876.64	694.90	592.68	822.44	718.29
Crystal system	Triclinic	Monoclinic	Orthorhombic	Monoclinic	Triclinic
Space group	*P-1*	*C2/c*	*Pna2₁*	*C2/c*	*P-1*
a/Å	10.0572(13)	25.103(11)	15.528(5)	29.933(9)	9.357(4)
b/Å	12.698(2)	13.921(6)	25.426(8)	9.062(3)	12.332(4)
c/Å	16.365(2)	10.351(5)	7.073(3)	24.805(7)	13.609(4)
A	83.638(16)	90	90	90	116.52(2)
B	74.068(16)	95.719(9)	90	98.190(8)	95.79(4)
Γ	73.326(14)	90	90	90	93.31(3)
U/A^3	1924.0(5)	3599(3)	2792.4(15)	6660(3)	1389.4(9)
Z	2	4	4	8	2
μ/mm^{-1}	10.906	3.871	4.858	45.301	54.147
Reflections collected	14862	13723	20858	25008	10673
Independent reflections	6713	3156	4811	5845	4848
R_{int}	0.0763	0.1035	0.1244	0.1048	0.0759
R1	0.0604	0.0699	0.0897	0.0840	0.0943
wR2 [I > 2σ(I)]	0.1269	0.1994	0.1489	0.2190	0.2675

3. ^{1}H and ^{13}C NMR spectra of compounds 1-17, 19, 20-29, and ^{1}H, ^{13}C NMR and ^{77}Se spectra for compounds 30 and 40

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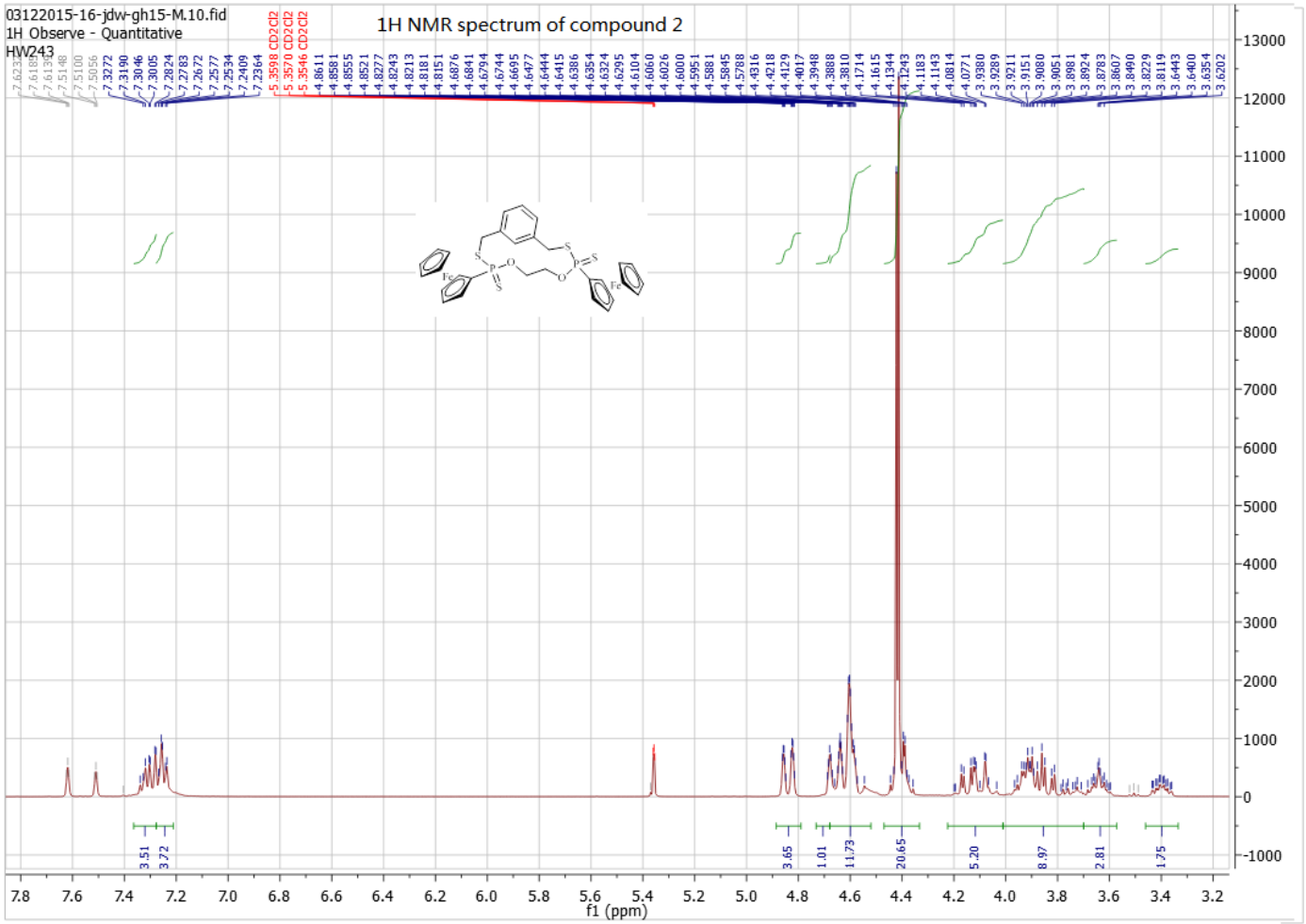
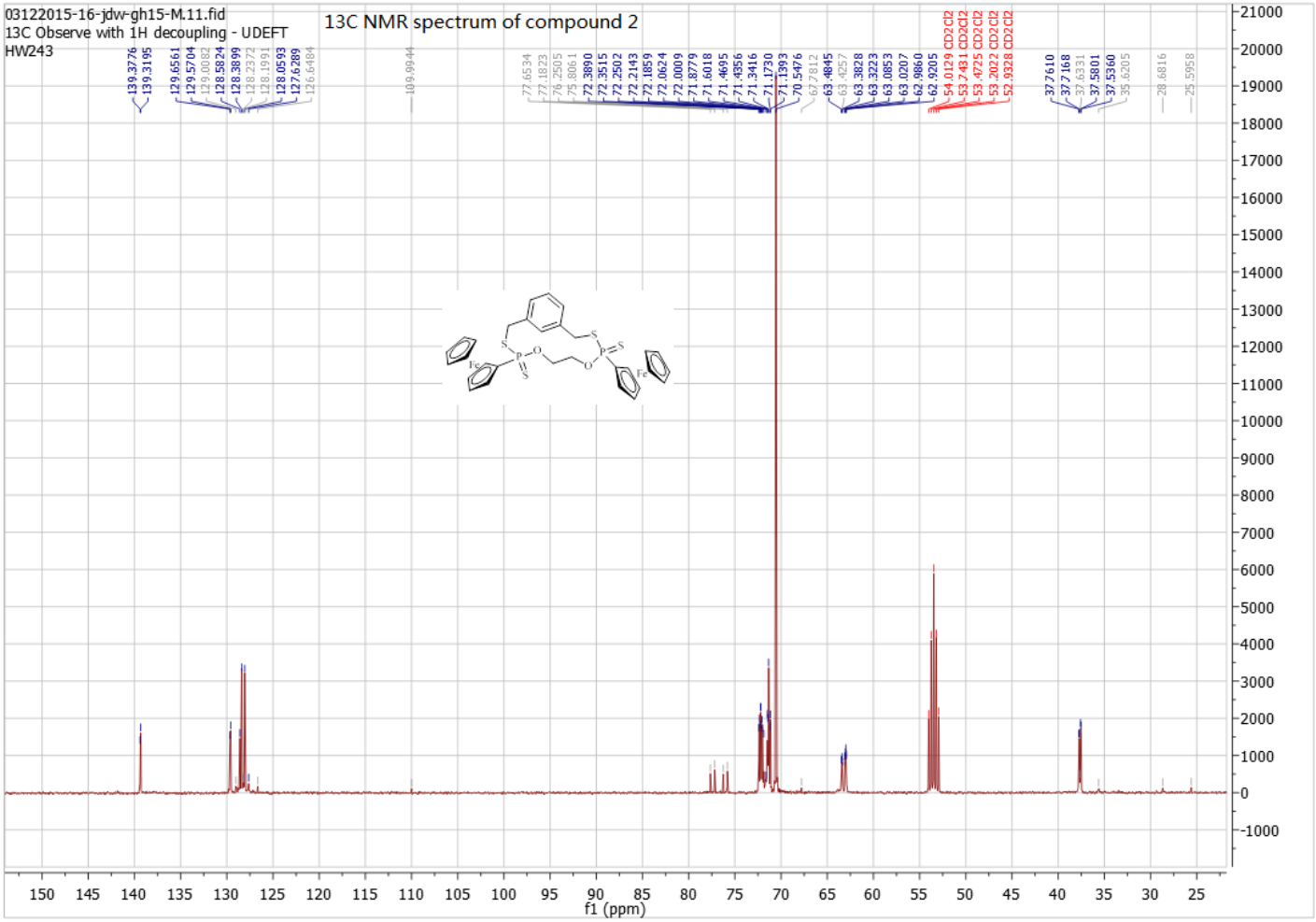
13C Observe with 1H decoupling - UDEFT
HW242
13C NMR spectrum of compound 1
f1 (ppm)


03122015-16-jdw-gh15-M.10.fid
1H Observe - Quantitative
HW243
1H NMR spectrum of compound 2
f1 (ppm)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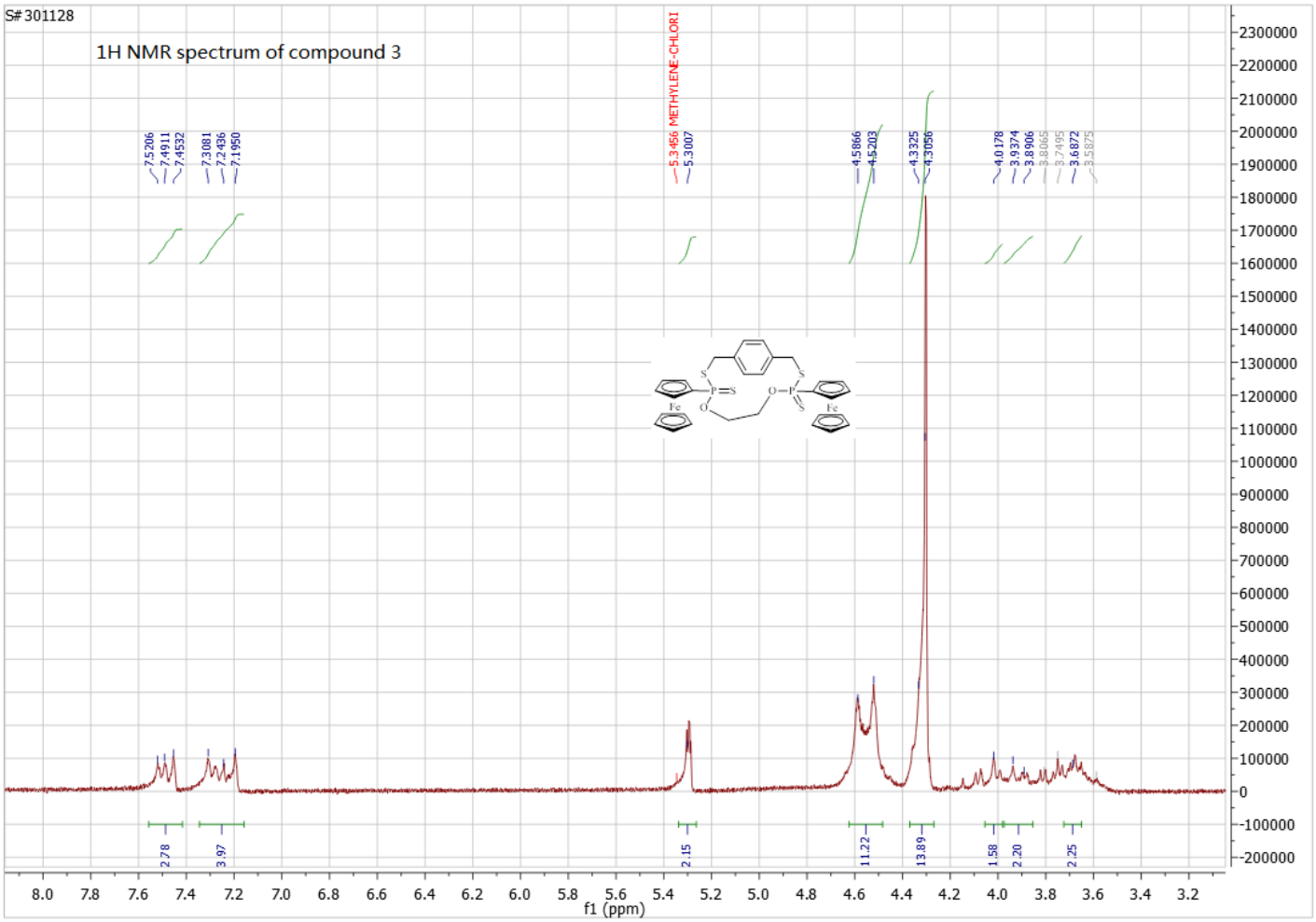
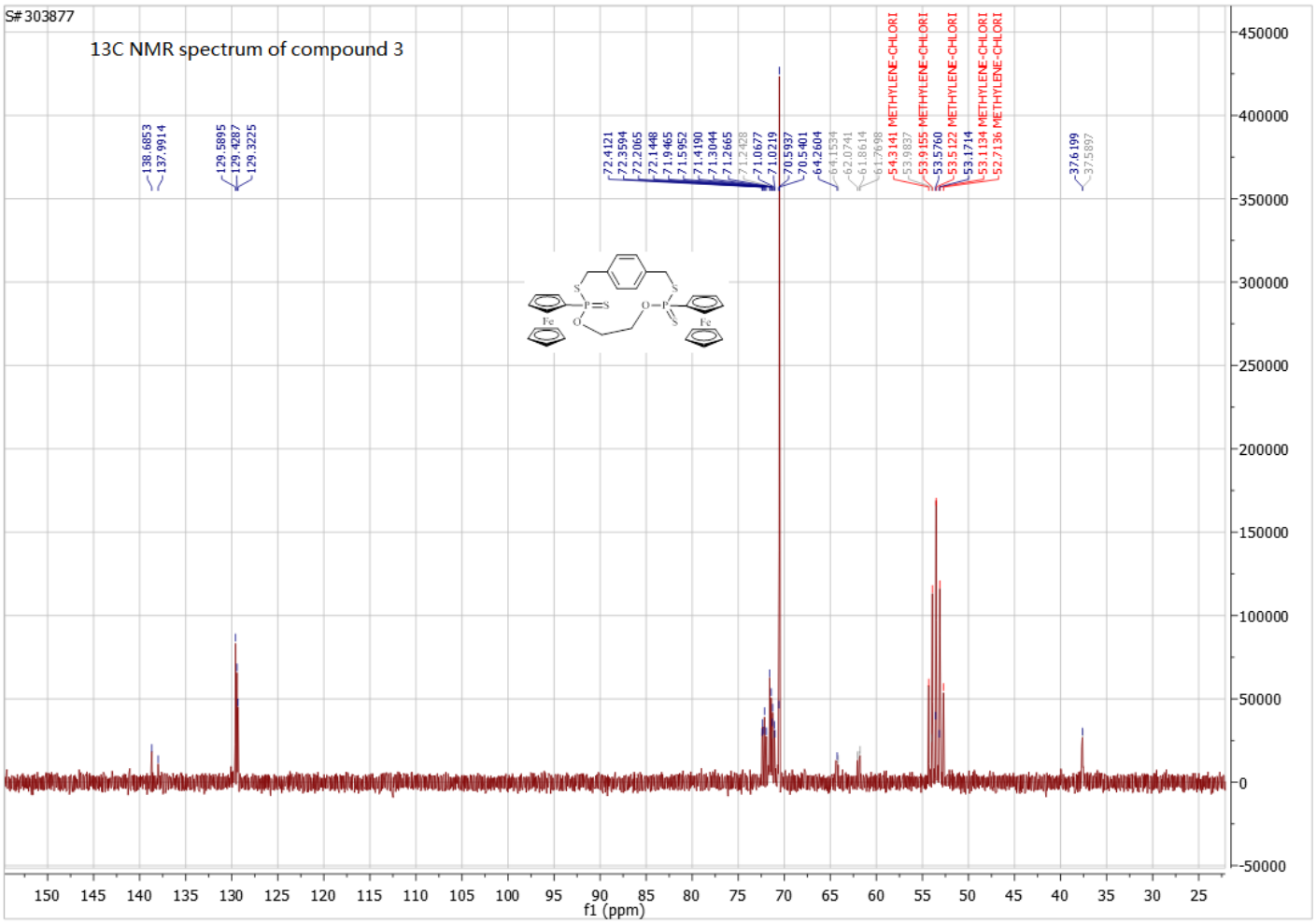
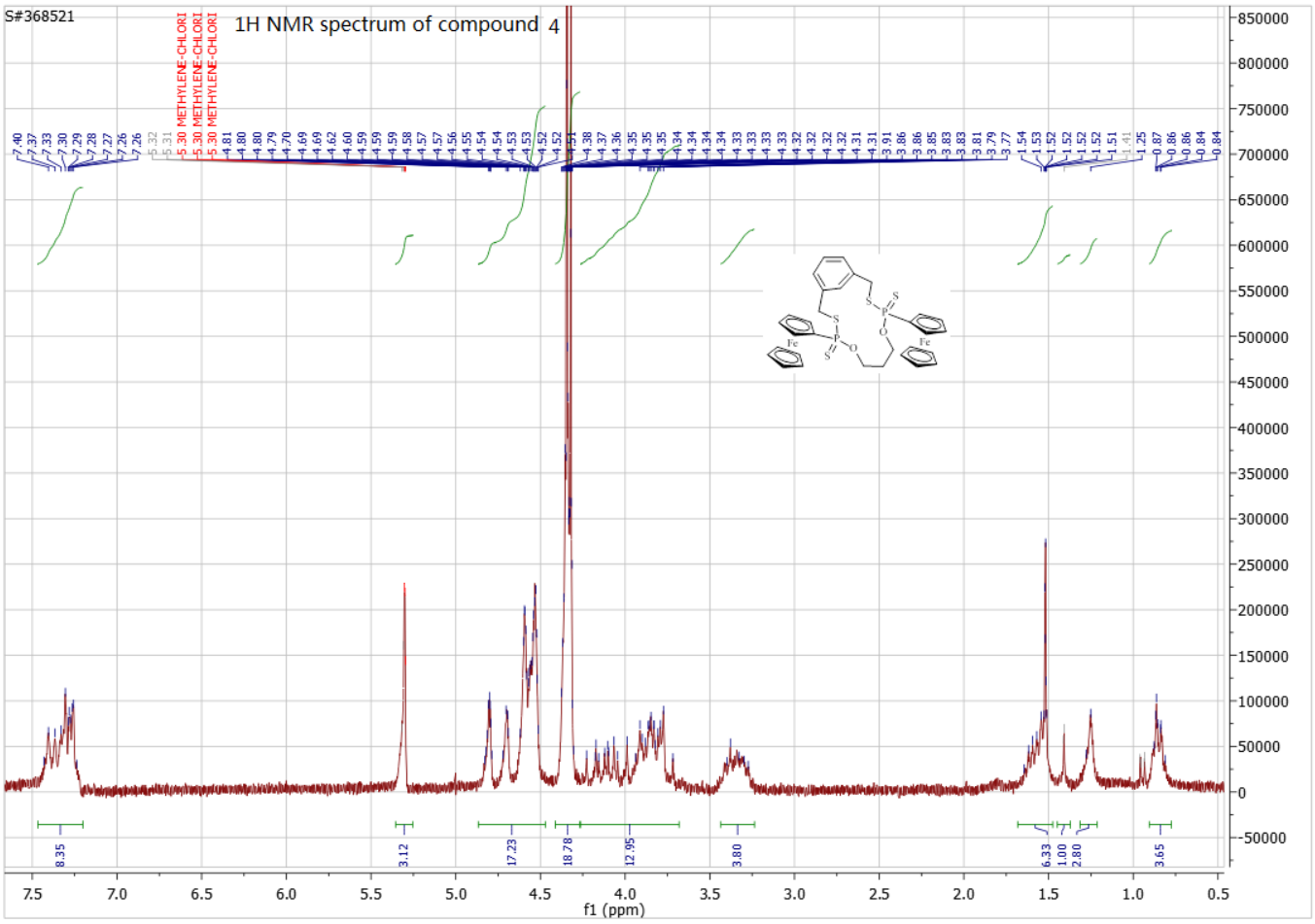
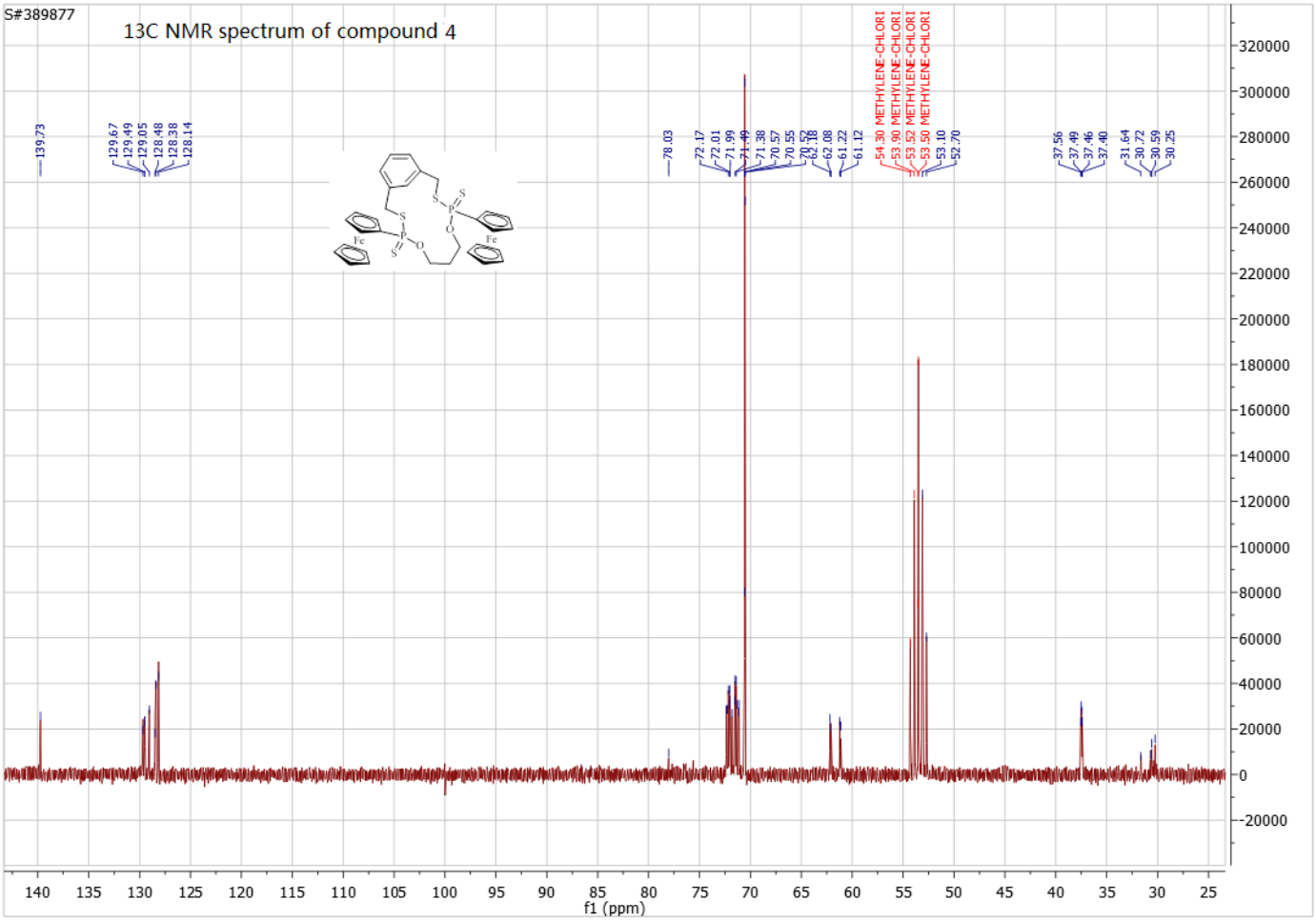
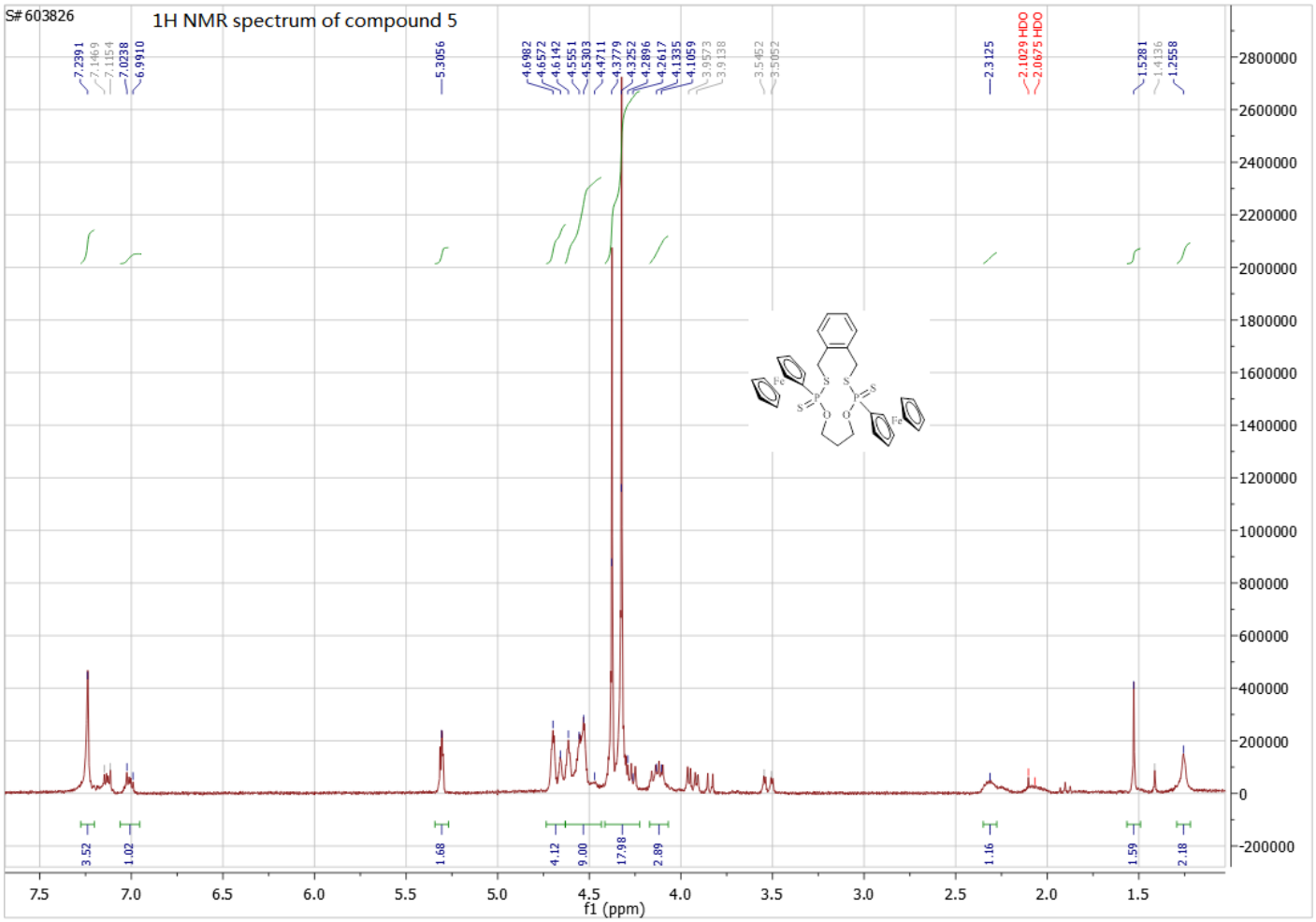
13C Observe with 1H decoupling - UDEFT
HW243
13C NMR spectrum of compound 2
f1 (ppm)

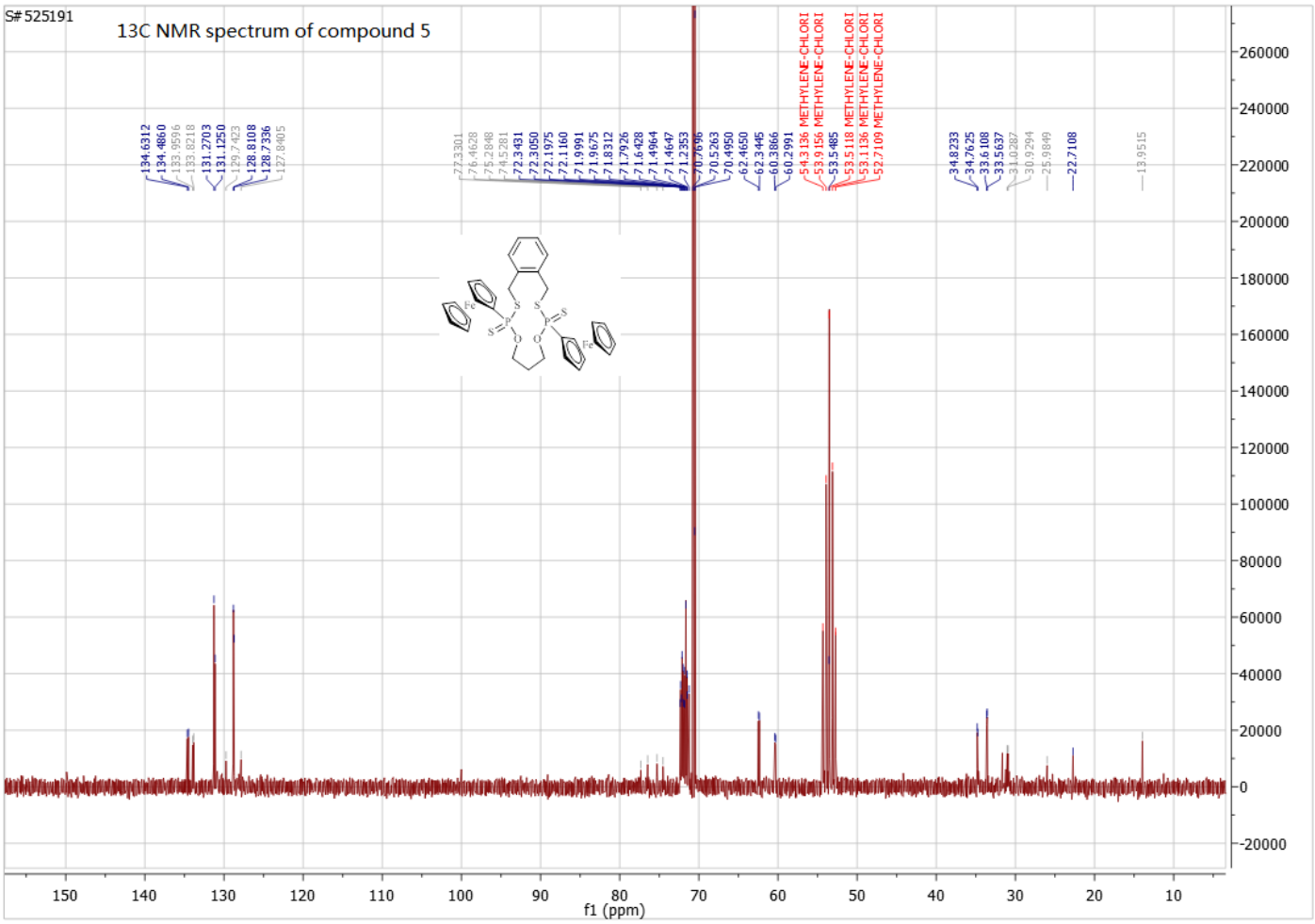

S# 301128
1H NMR spectrum of compound 3
METHYLENE-CHLORI
7.5206
7.4911
7.4532
7.3081
7.2436
7.1950
5.3466
5.3007
4.5866
4.5203
4.3325
4.3056
4.0178
3.9374
3.8906
3.8065
3.7495
3.6872
3.5875
2.78
3.97
2.15
11.22
13.89
1.58
2.20
2.25
f1 (ppm)


S# 303877
13C NMR spectrum of compound 3
138.6853
137.9914
129.5895
129.4287
129.3225
72.4121
72.3594
72.2065
72.1448
71.9465
71.5952
71.4190
71.3044
71.2665
71.2428
71.0677
71.0219
70.5937
70.5401
64.2604
64.1534
62.0741
61.8614
61.7698
54.3141 METHYLENE-CHLORI
53.9837
53.9155 METHYLENE-CHLORI
53.5760
53.5122 METHYLENE-CHLORI
53.1714
53.1134 METHYLENE-CHLORI
52.7136 METHYLENE-CHLORI
37.6199
37.5897
Fe
Fe
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25
f1 (ppm)


S#368521
1H NMR spectrum of compound 4
METHYLENE-CHLORI
f1 (ppm)


S#389877
13C NMR spectrum of compound 4
139.73
129.67
129.49
129.05
128.48
128.38
128.14
78.03
72.17
72.01
71.99
71.49
71.38
70.57
70.55
70.52
62.18
62.08
61.22
61.12
54.30 METHYLENE-CHLORI
53.90 METHYLENE-CHLORI
53.52 METHYLENE-CHLORI
53.50 METHYLENE-CHLORI
53.10
52.70
37.56
37.49
37.46
37.40
31.64
30.72
30.59
30.25
Fe
Fe
320000
300000
280000
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25
f1 (ppm)


S# 603826
1H NMR spectrum of compound 5
7.2391
7.1469
7.1154
7.0238
6.9910
5.3056
4.6982
4.6572
4.6142
4.5551
4.5303
4.4711
4.3779
4.3252
4.2896
4.2617
4.1335
4.1059
3.9573
3.9138
3.5452
3.5052
2.3125
2.1029 HDO
2.0675 HDO
1.5281
1.4136
1.2558
3.52
1.02
1.68
4.12
9.00
17.98
2.89
1.16
1.59
2.18
f1 (ppm)


S# 525191
13C NMR spectrum of compound 5
134.6312
134.4860
133.9596
133.8218
131.2703
131.1250
129.7423
128.8108
128.7336
127.8405
77.3301
76.4628
75.2848
74.5281
72.3431
72.3050
72.1975
72.1160
71.9991
71.9675
71.8312
71.7926
71.6428
71.4964
71.4647
71.2353
70.7696
70.5263
70.4950
62.4650
62.3445
60.3866
60.2991
54.3136 METHYLENE-CHLORI
53.9156 METHYLENE-CHLORI
53.5485
53.5118 METHYLENE-CHLORI
53.1136 METHYLENE-CHLORI
52.7109 METHYLENE-CHLORI
34.8233
34.7625
33.6108
33.5637
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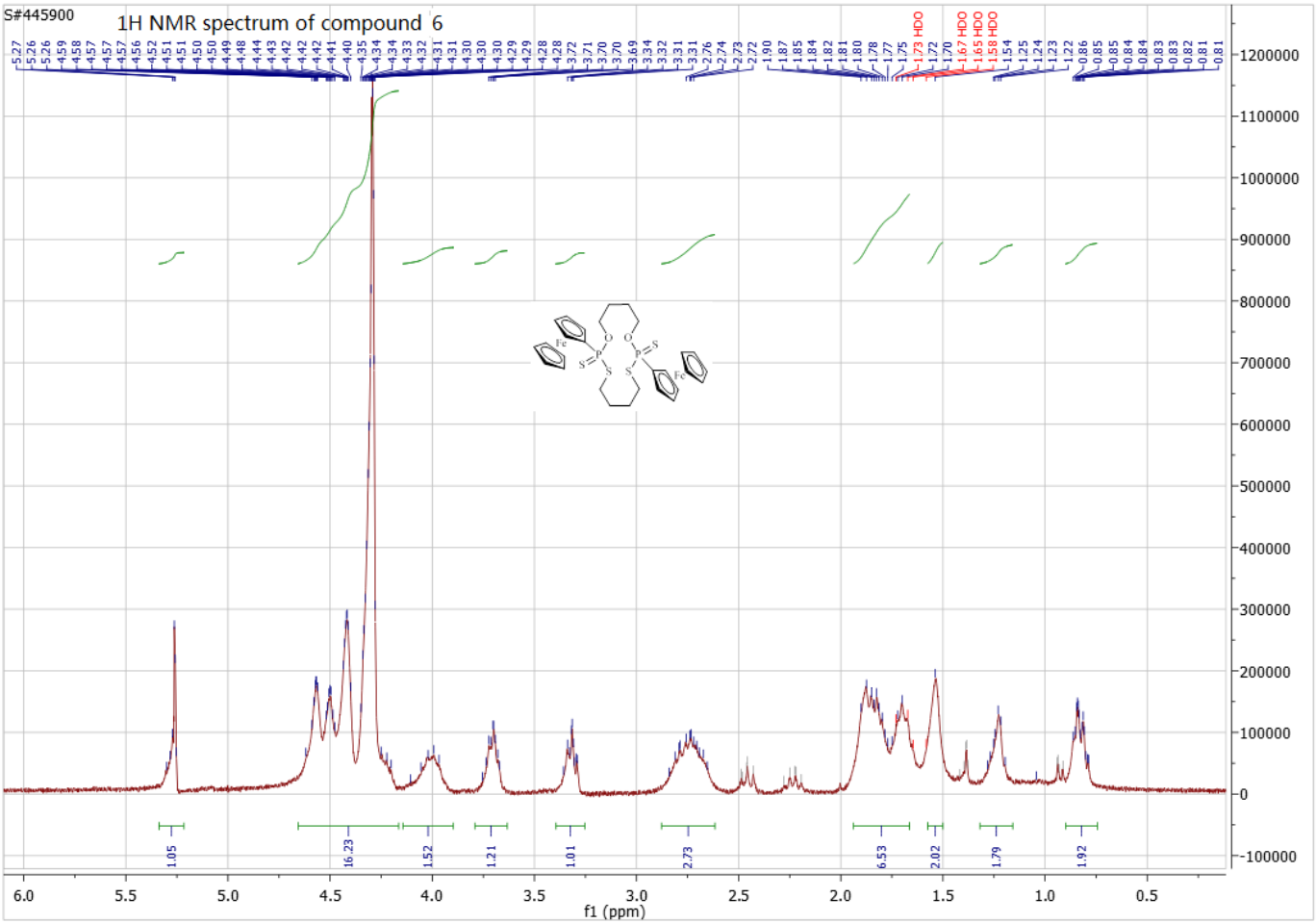
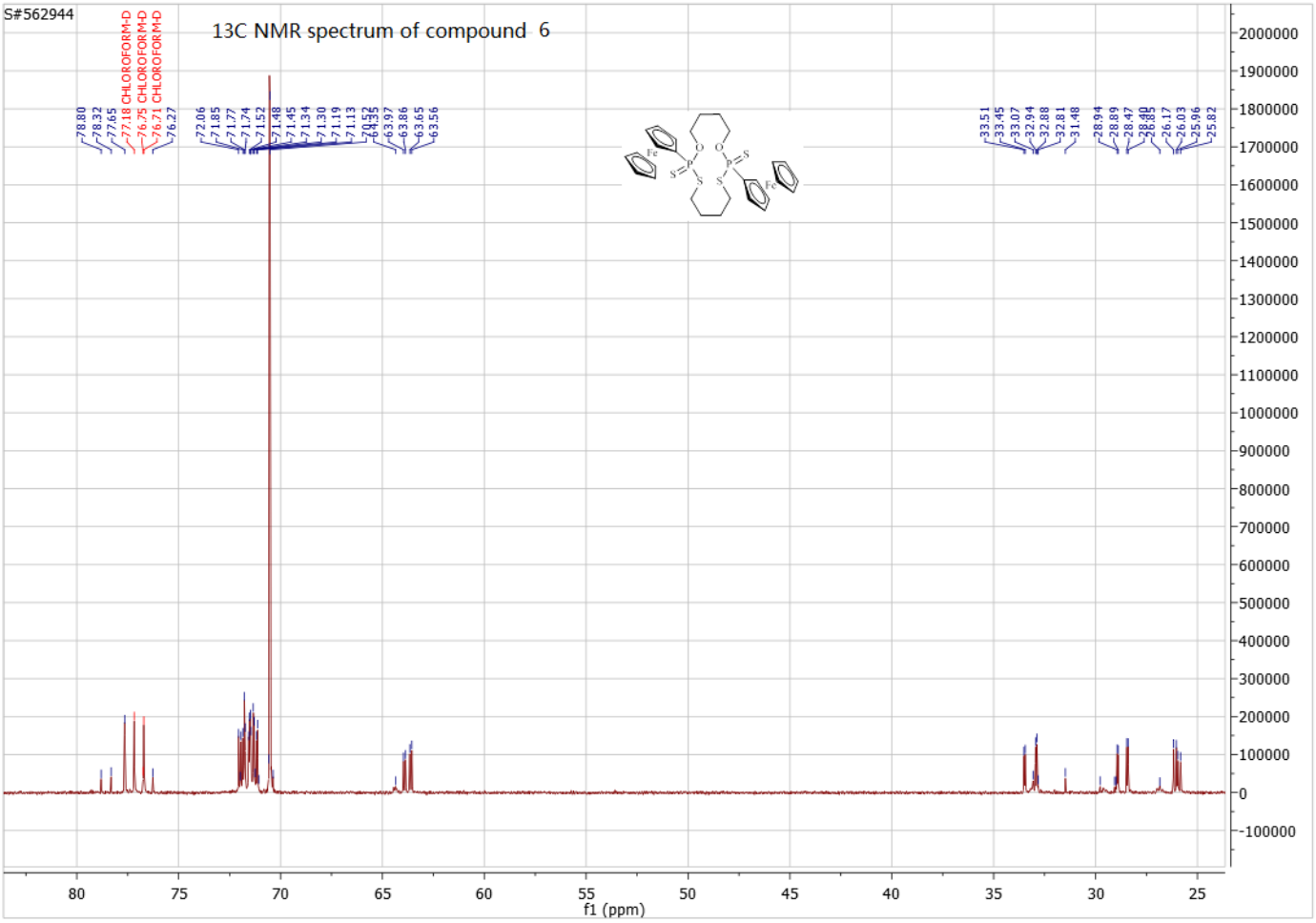
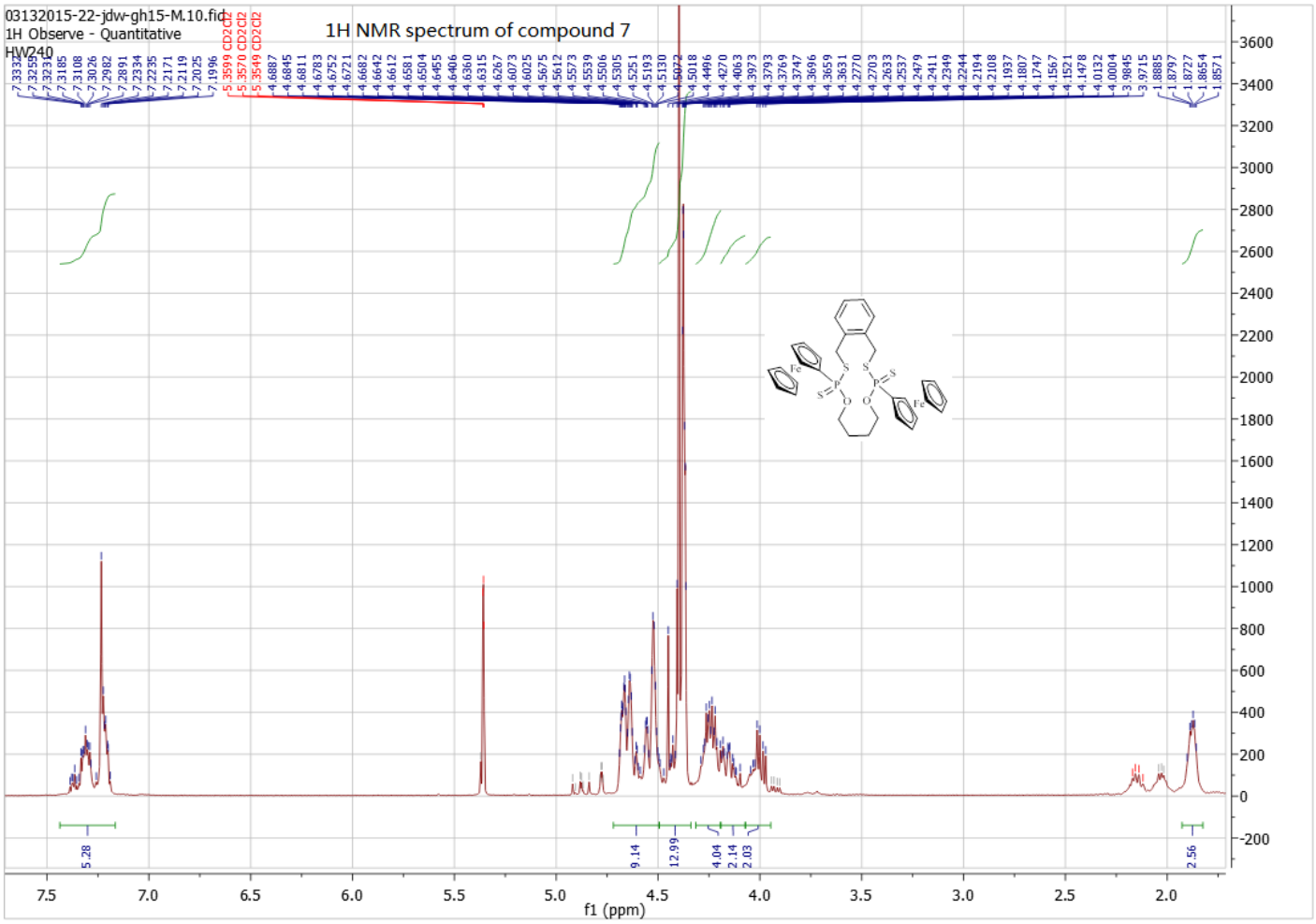
25.9849
22.7108
13.9515
f1 (ppm)


S#445900
1H NMR spectrum of compound 6
HDO
HDO
HDO
1.05
16.23
1.52
1.21
1.01
2.73
6.53
2.02
1.79
1.92
f1 (ppm)


S#562944
13C NMR spectrum of compound 6
78.80
78.32
77.65
77.18 CHLOROFORM-D
76.75 CHLOROFORM-D
76.71 CHLOROFORM-D
76.27
72.06
71.85
71.77
71.74
71.52
71.48
71.45
71.34
71.30
71.19
71.13
70.52
70.35
63.97
63.86
63.65
63.56
33.51
33.45
33.07
32.94
32.88
32.81
31.48
28.94
28.89
28.47
28.40
26.85
26.17
26.03
25.96
25.82
2000000
1900000
1800000
1700000
1600000
1500000
1400000
1300000
1200000
1100000
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
-100000
80
75
70
65
60
55
50
45
40
35
30
25
f1 (ppm)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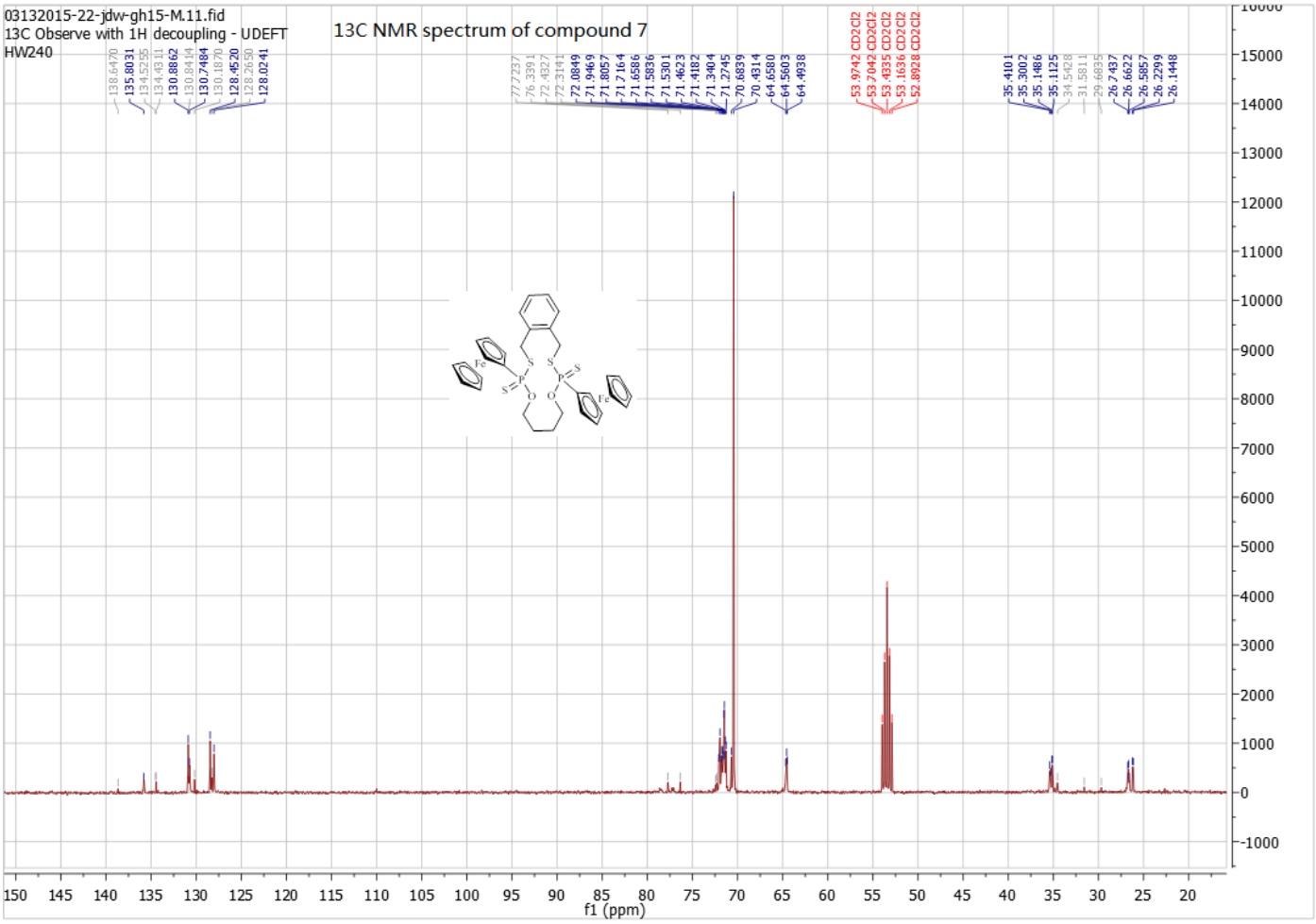
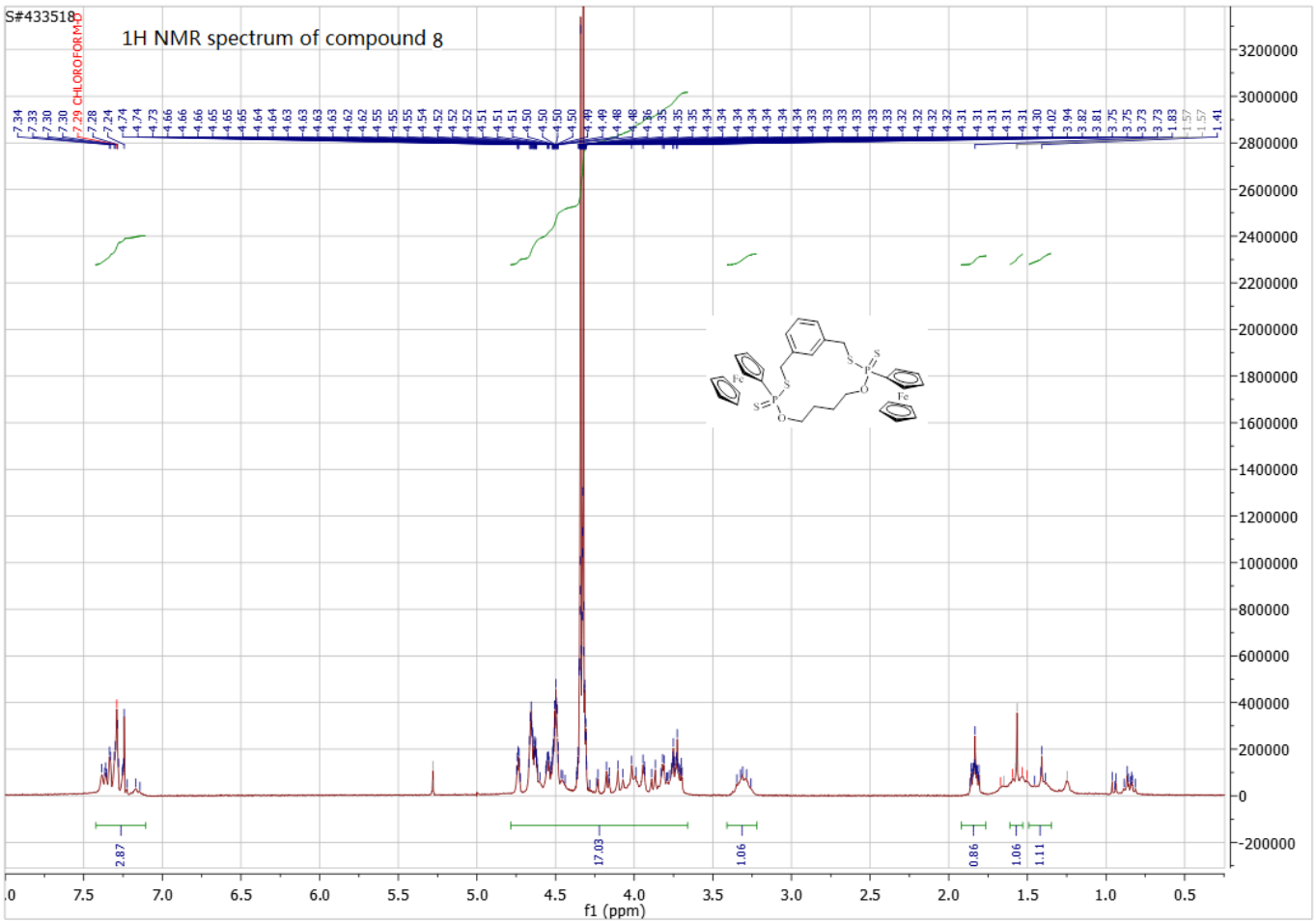
1H Observe - Quantitative
HW240
1H NMR spectrum of compound 7
f1 (ppm)


03132015-22-jdw-gh15-M.11.fid
13C Observe with 1H decoupling - UDEFT
HW240
13C NMR spectrum of compound 7
f1 (ppm)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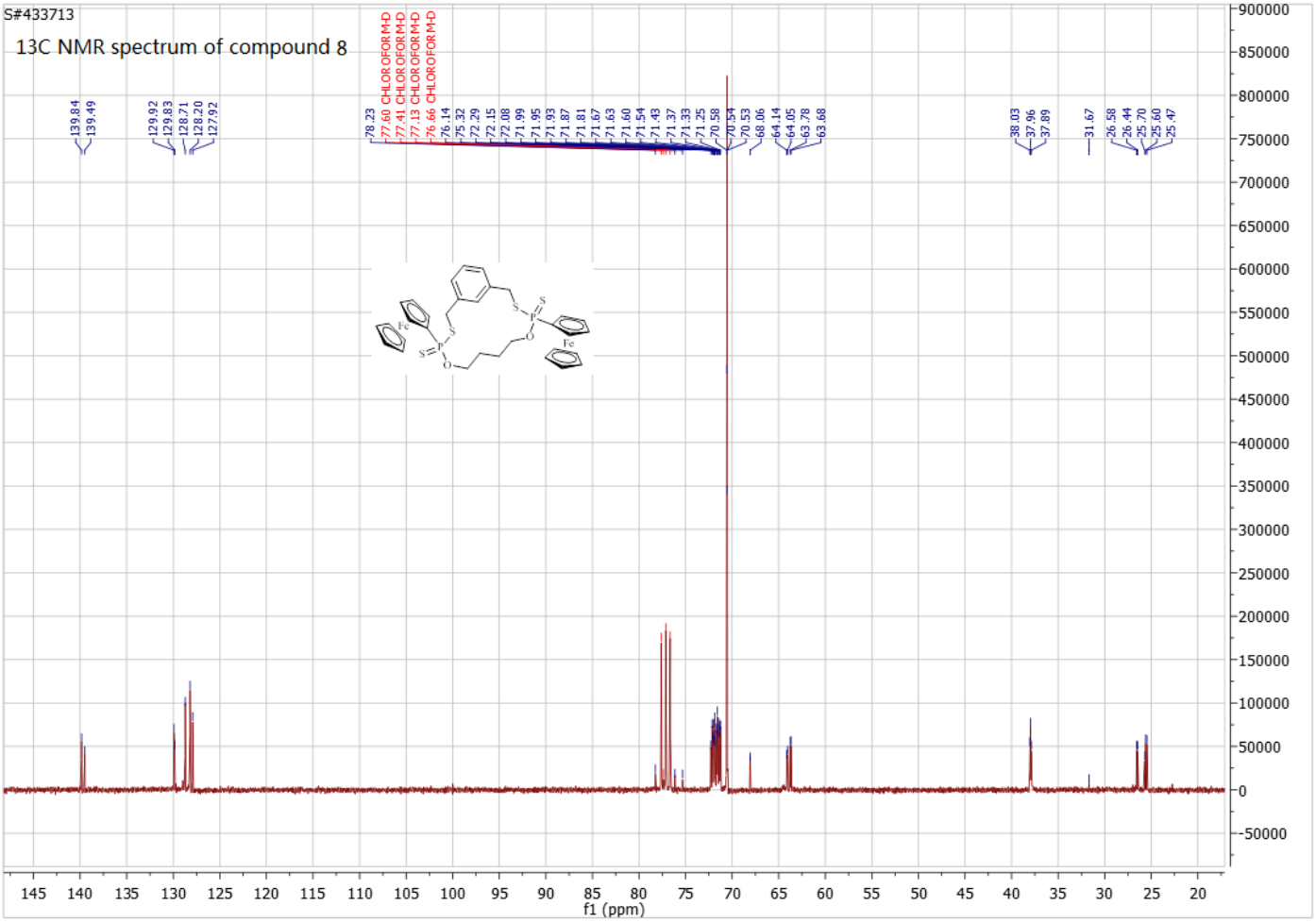
1H NMR spectrum of compound 8
CHLOROFORM-D
f1 (ppm)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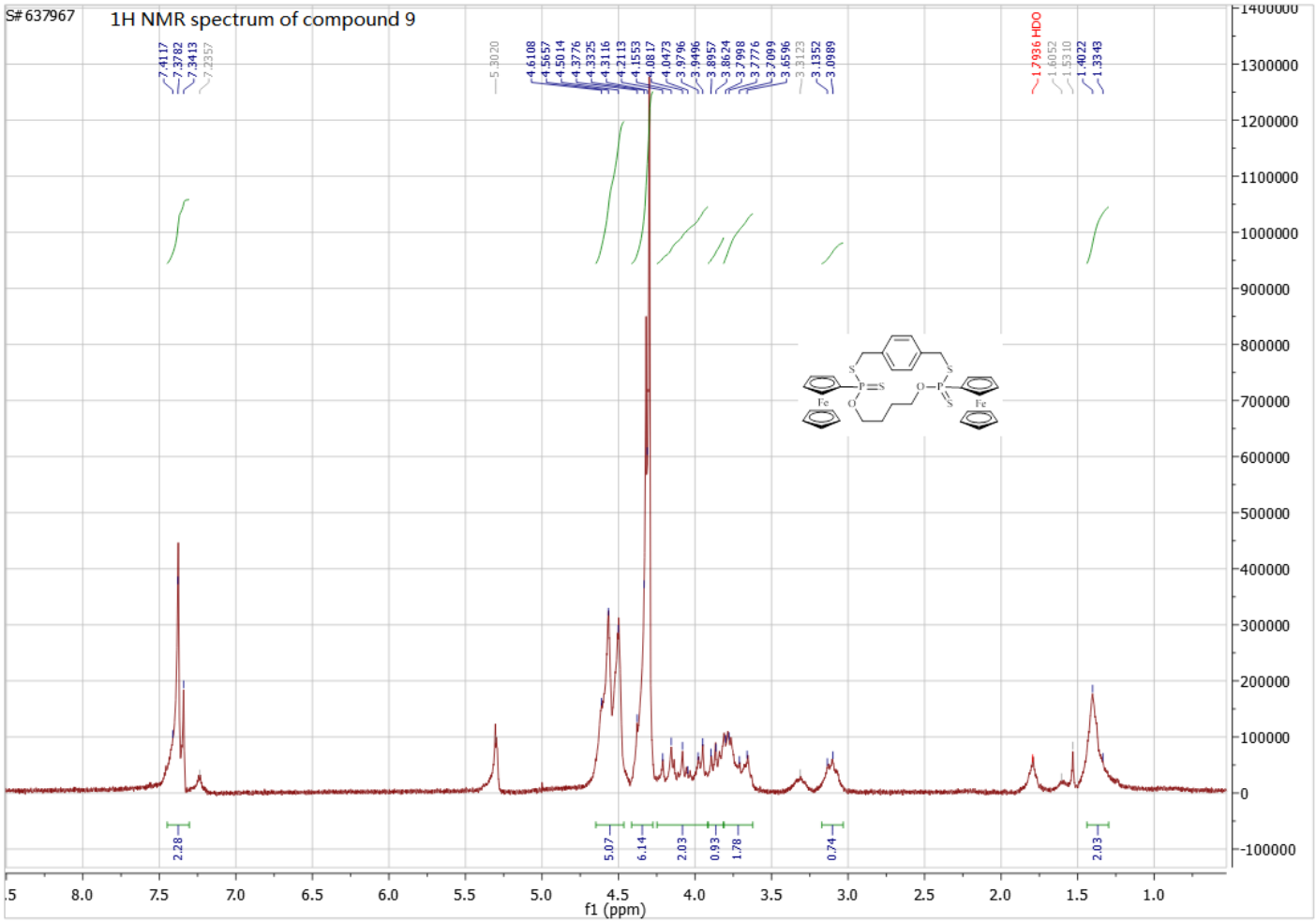
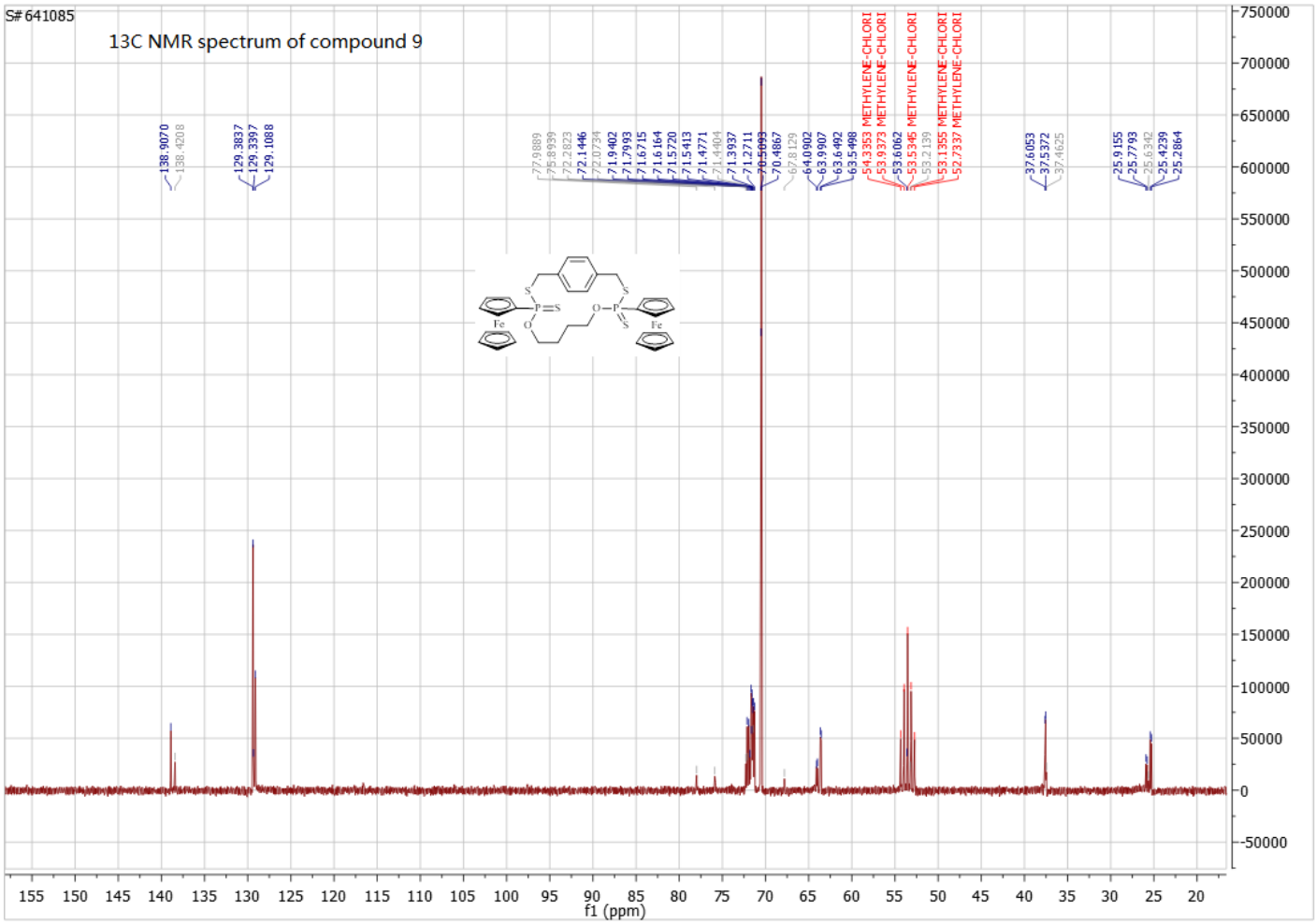
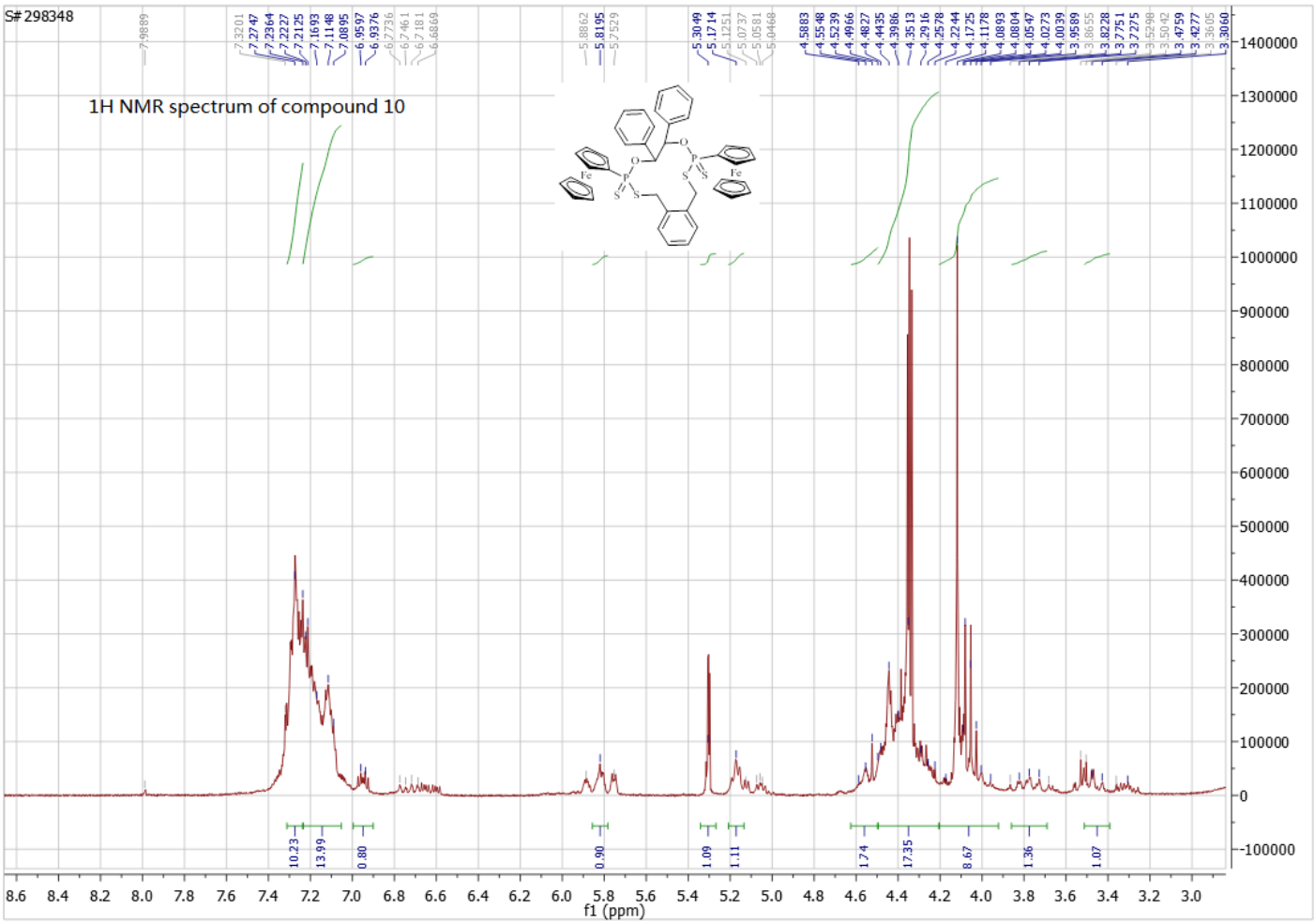
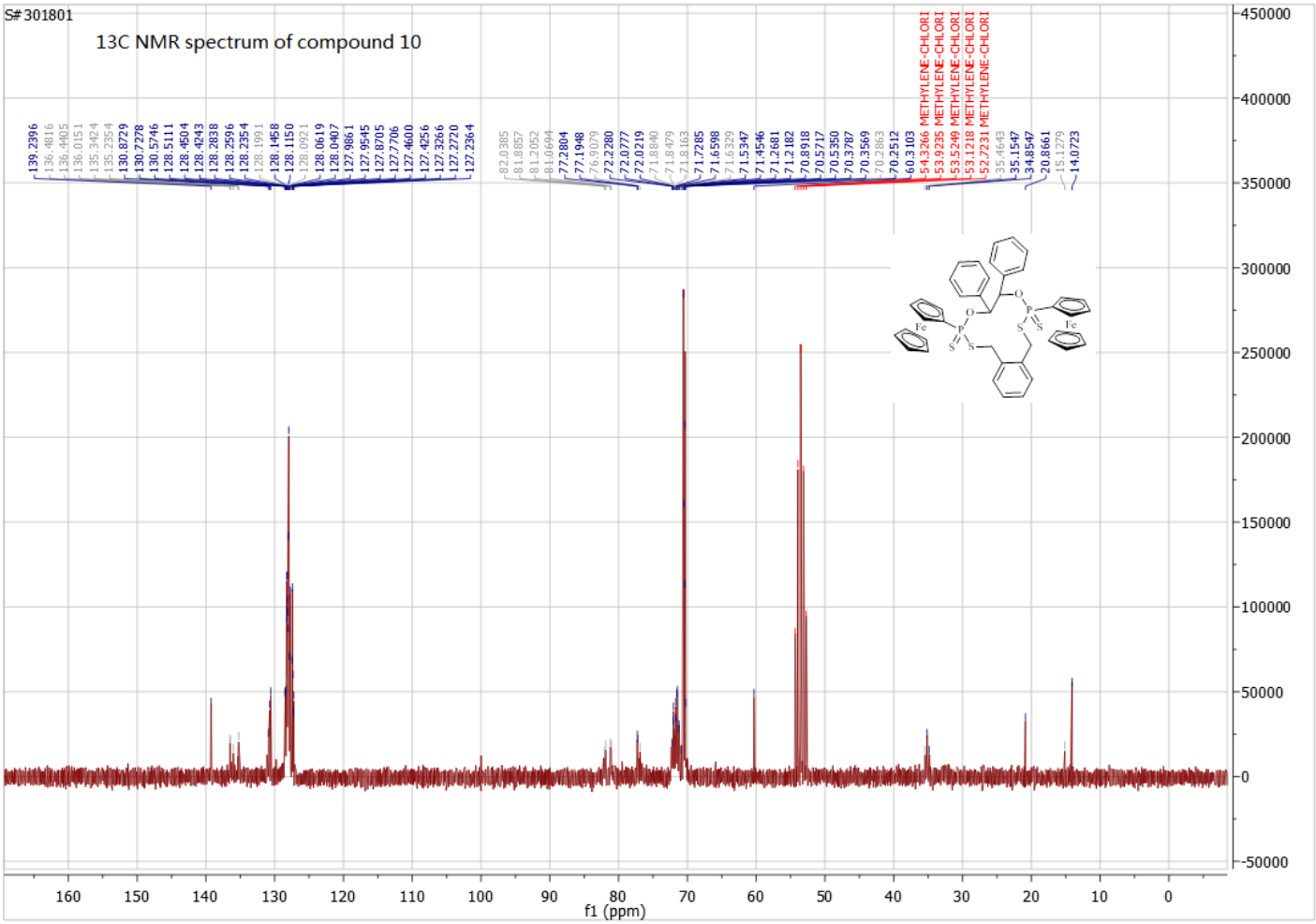
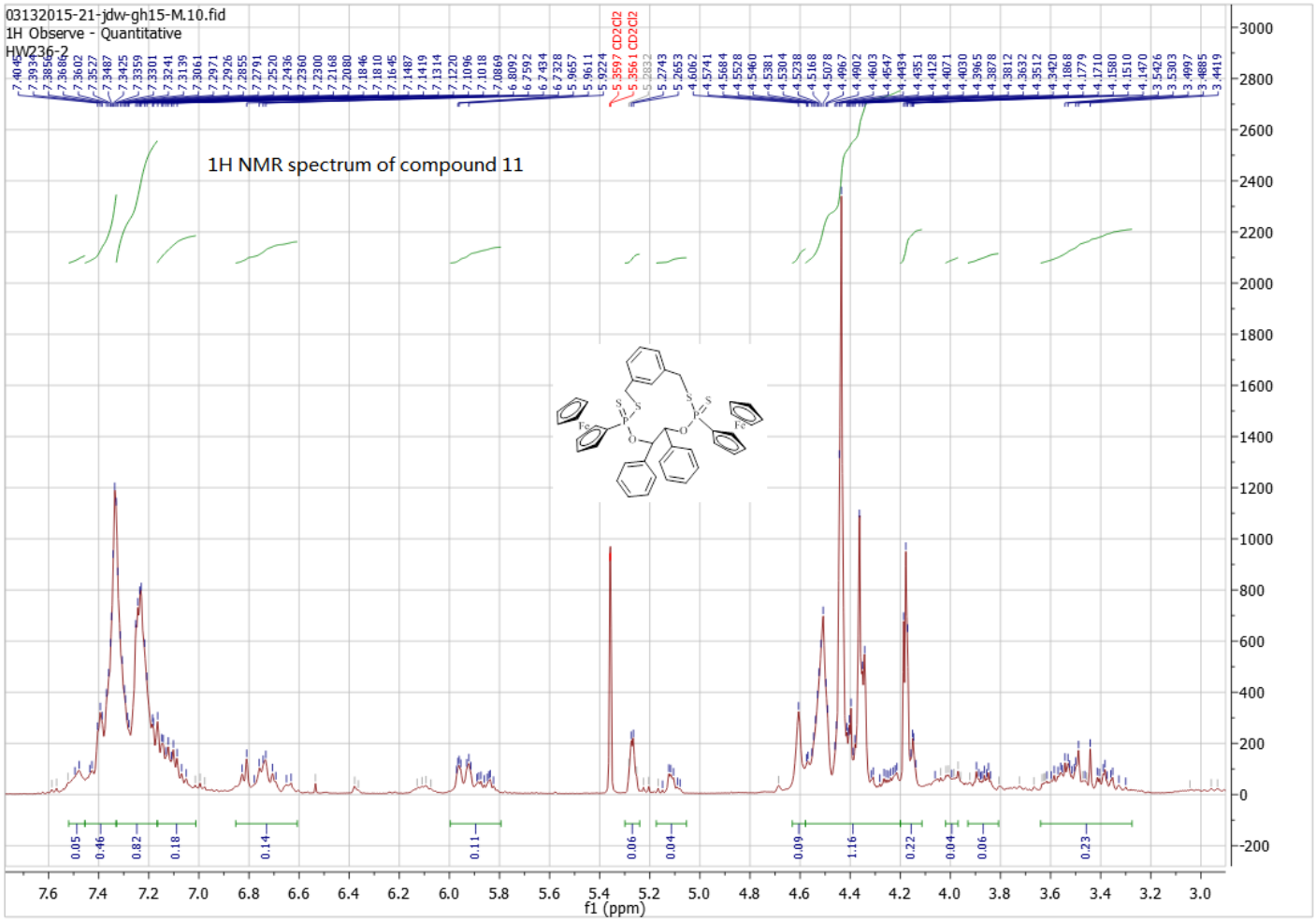
13C NMR spectrum of compound 8
f1 (ppm)

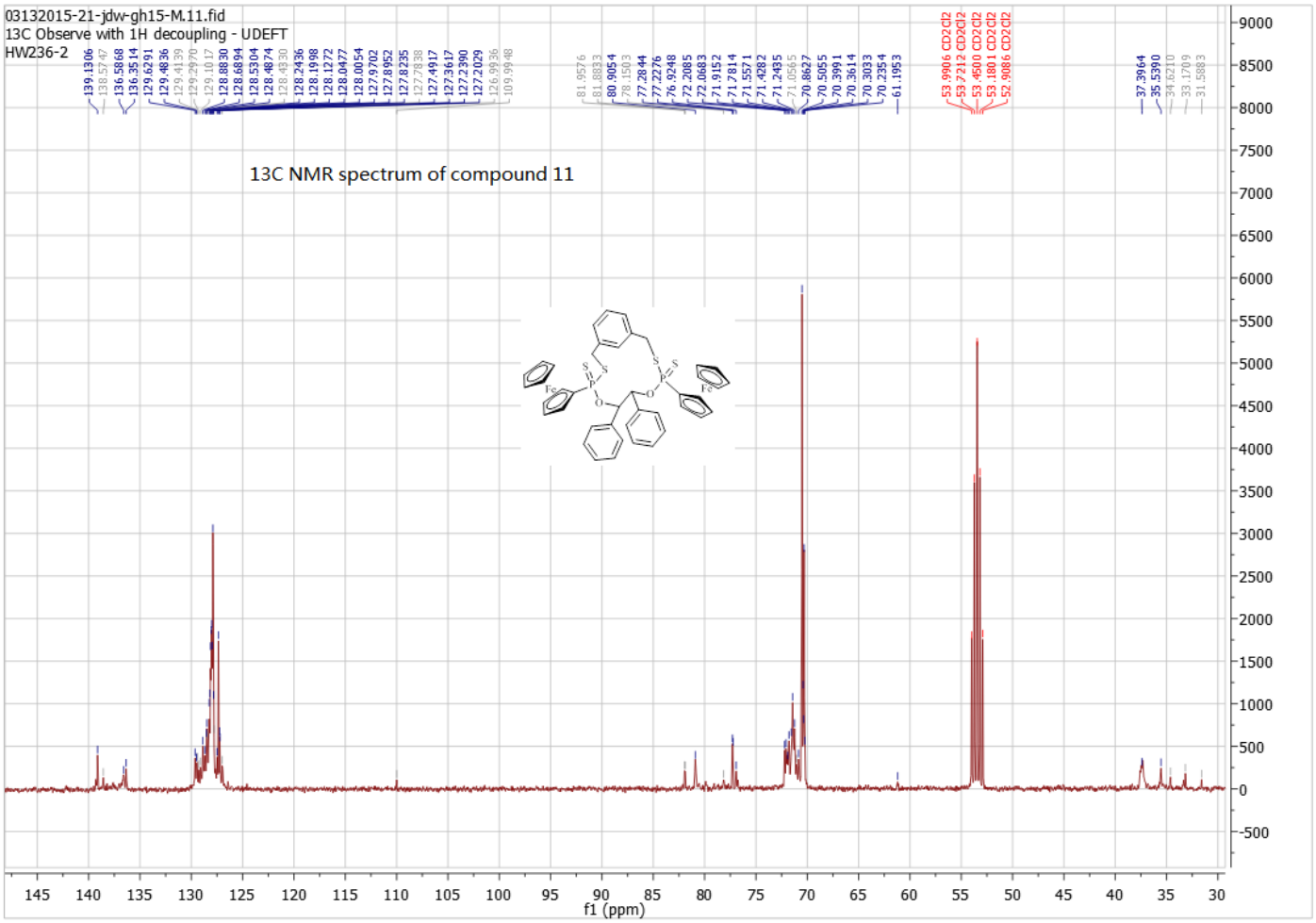

S#637967
1H NMR spectrum of compound 9
7.4117
7.3782
7.3413
7.2357
5.3020
4.6108
4.5657
4.5014
4.3776
4.3325
4.3116
4.2113
4.1553
4.0817
4.0473
3.9796
3.9496
3.8957
3.8624
3.7998
3.7776
3.7099
3.6596
3.3123
3.1352
3.0989
1.7936 HDO
1.6052
1.5310
1.4022
1.3343
2.28
5.07
6.14
2.03
0.93
1.78
0.74
2.03
f1 (ppm)

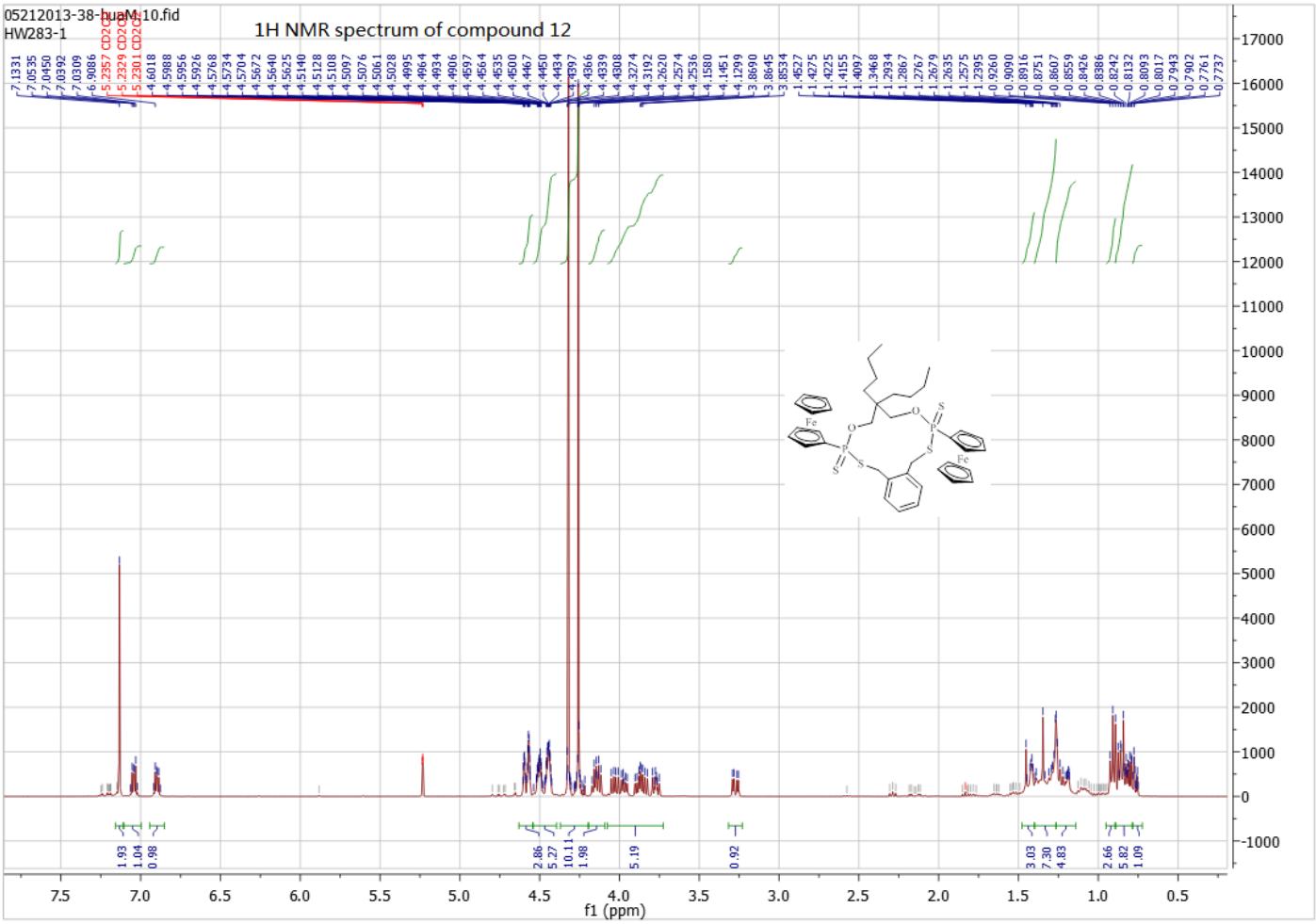

S# 641085
13C NMR spectrum of compound 9
f1 (ppm)

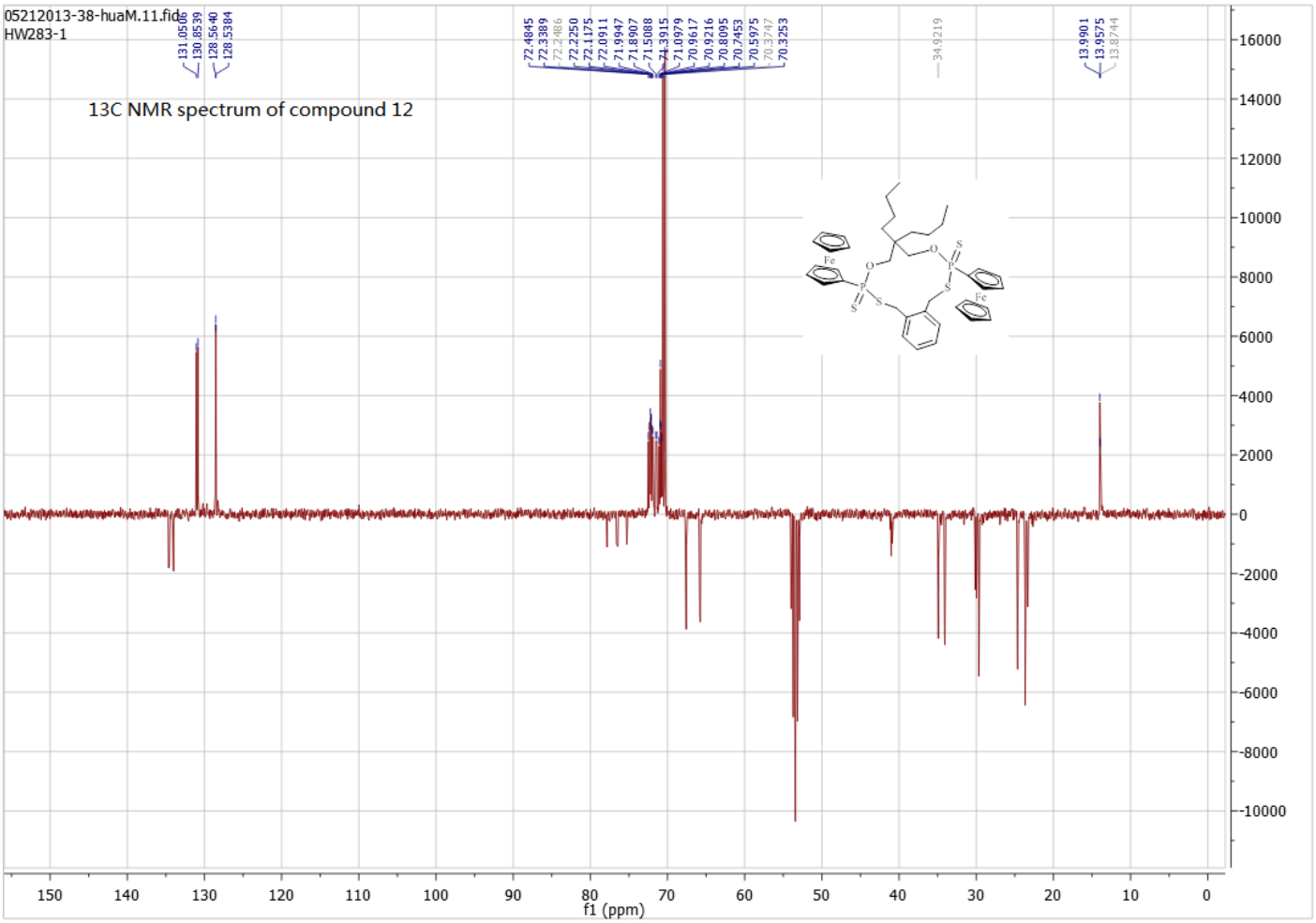

S# 298348
1H NMR spectrum of compound 10
f1 (ppm)

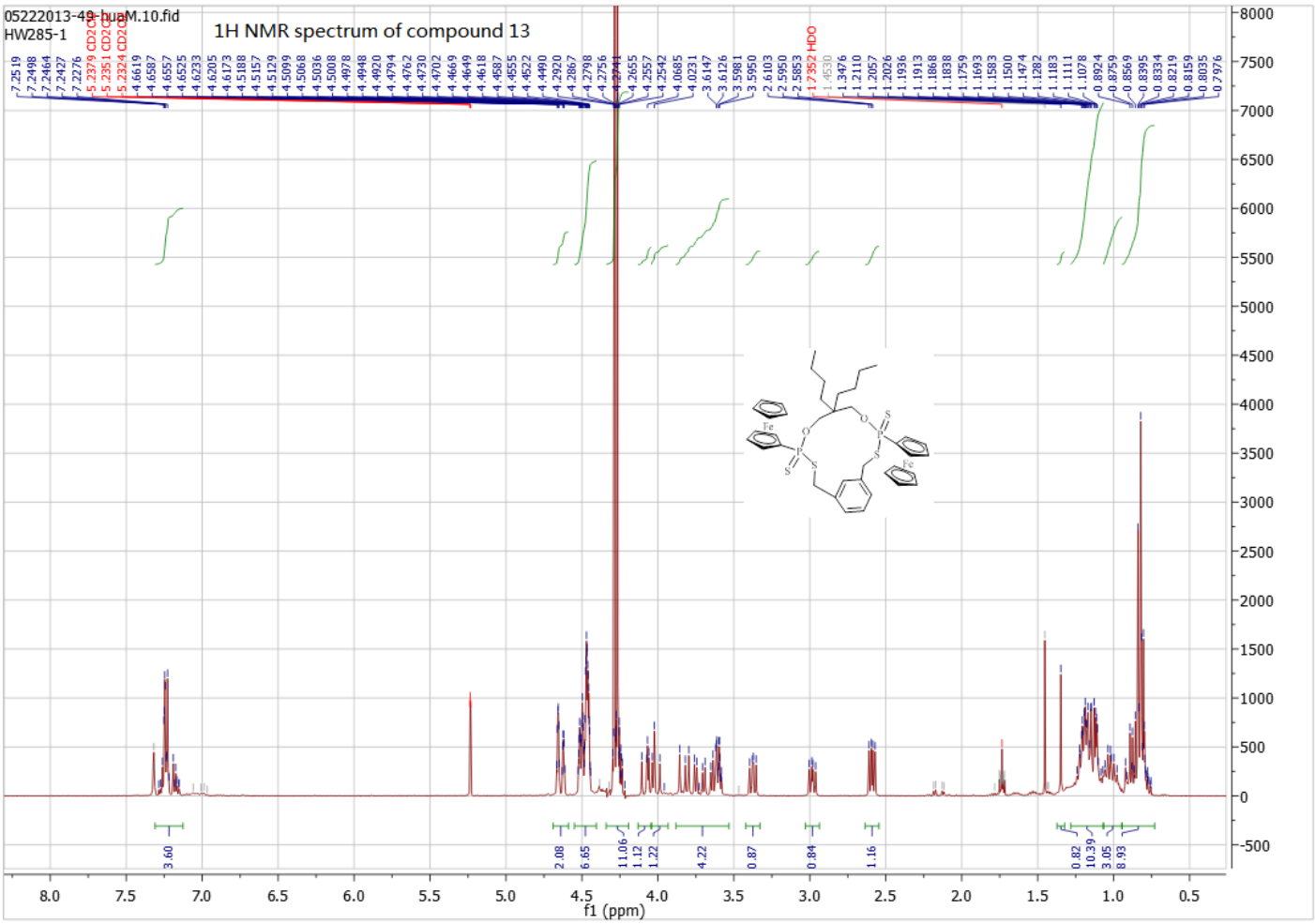

S# 301801
13C NMR spectrum of compound 10
f1 (ppm)

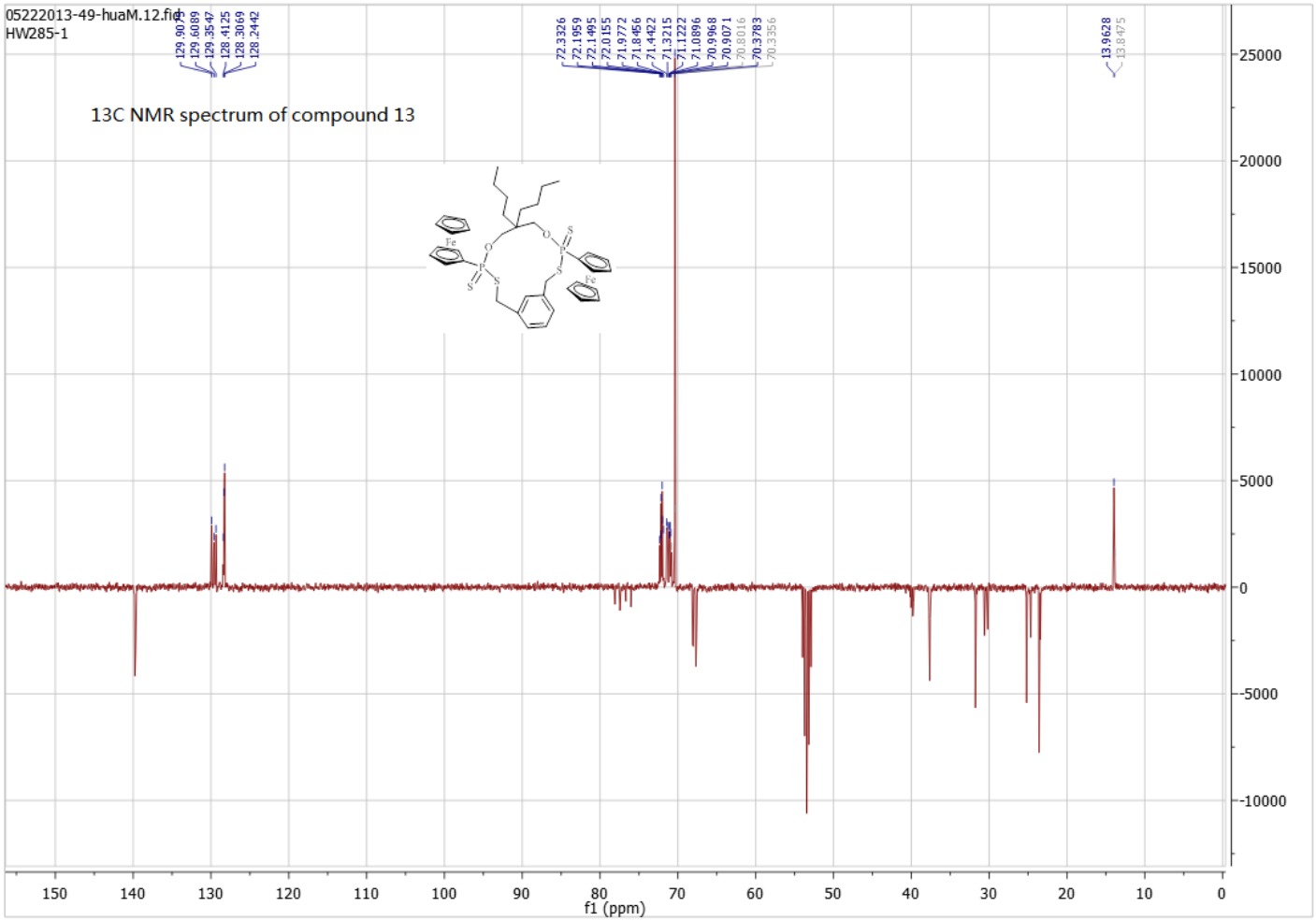

03132015-21-jdw-gh15-M.10.fid
1H Observe - Quantitative
HW236-2
1H NMR spectrum of compound 11
f1 (ppm)

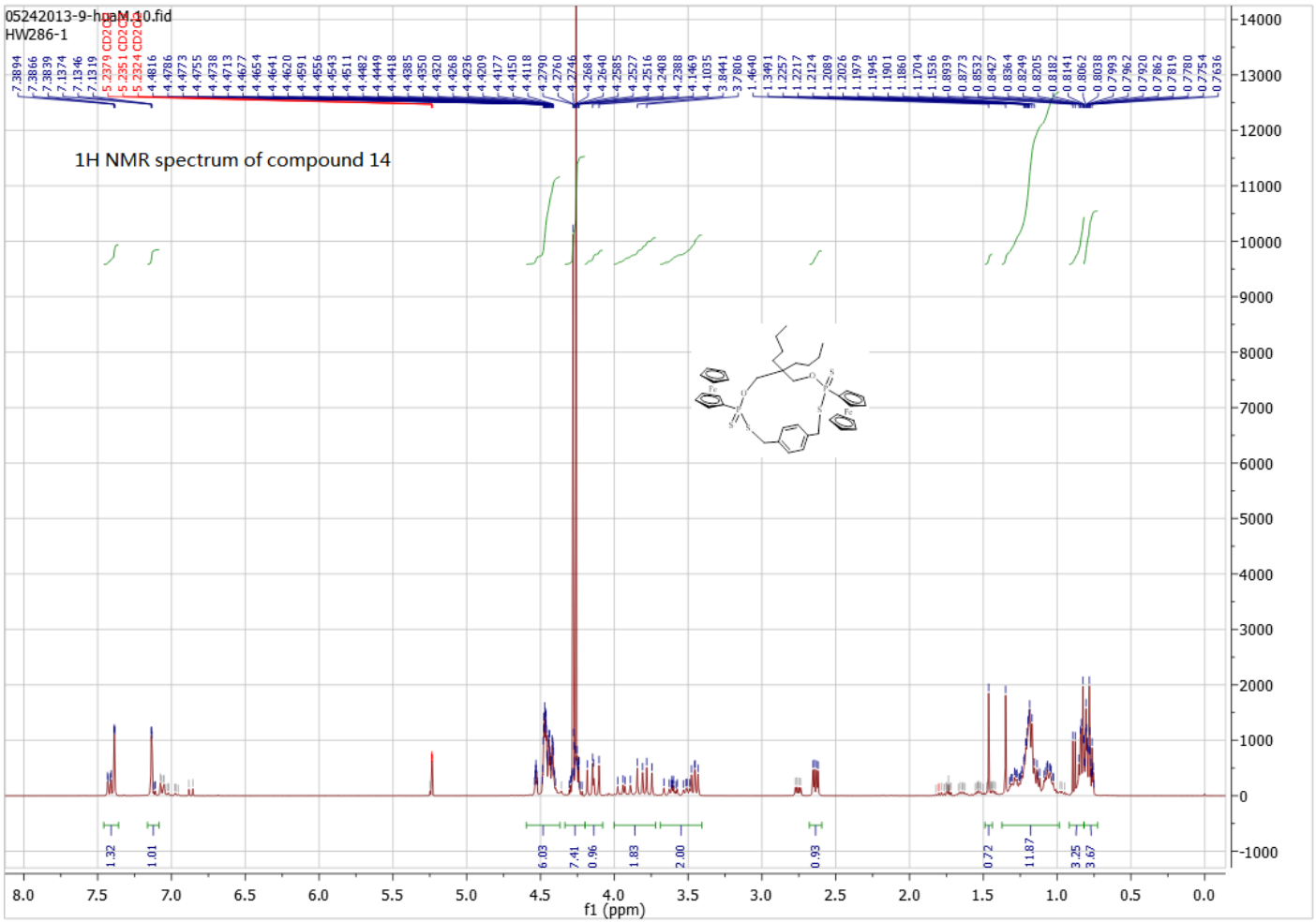

03132015-21-jdw-gh15-M.11.fid
13C Observe with 1H decoupling - UDEFT
HW236-2
13C NMR spectrum of compound 11
f1 (ppm)

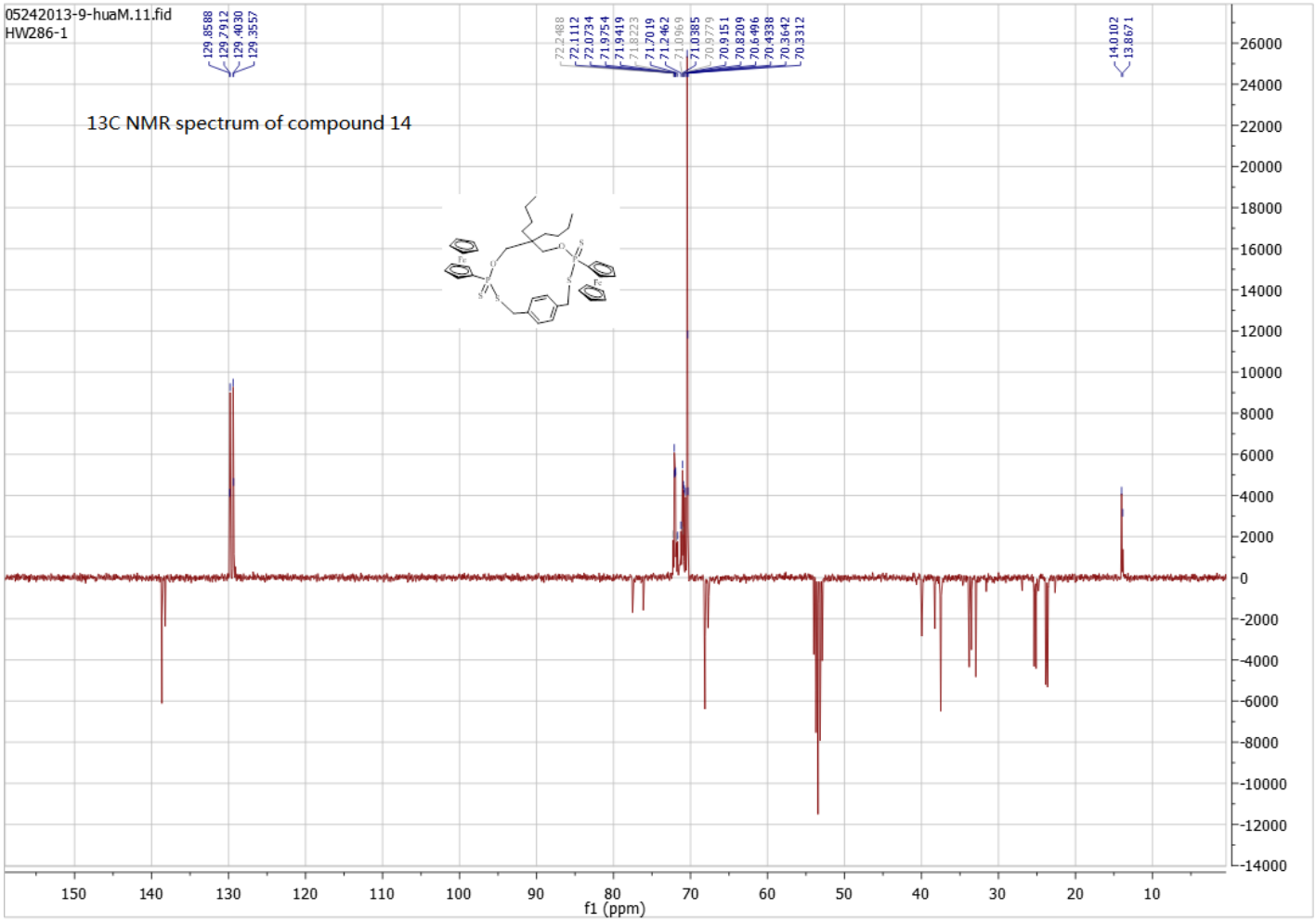

05212013-38-huaM-10.fid
HW283-1
1H NMR spectrum of compound 12
f1 (ppm)

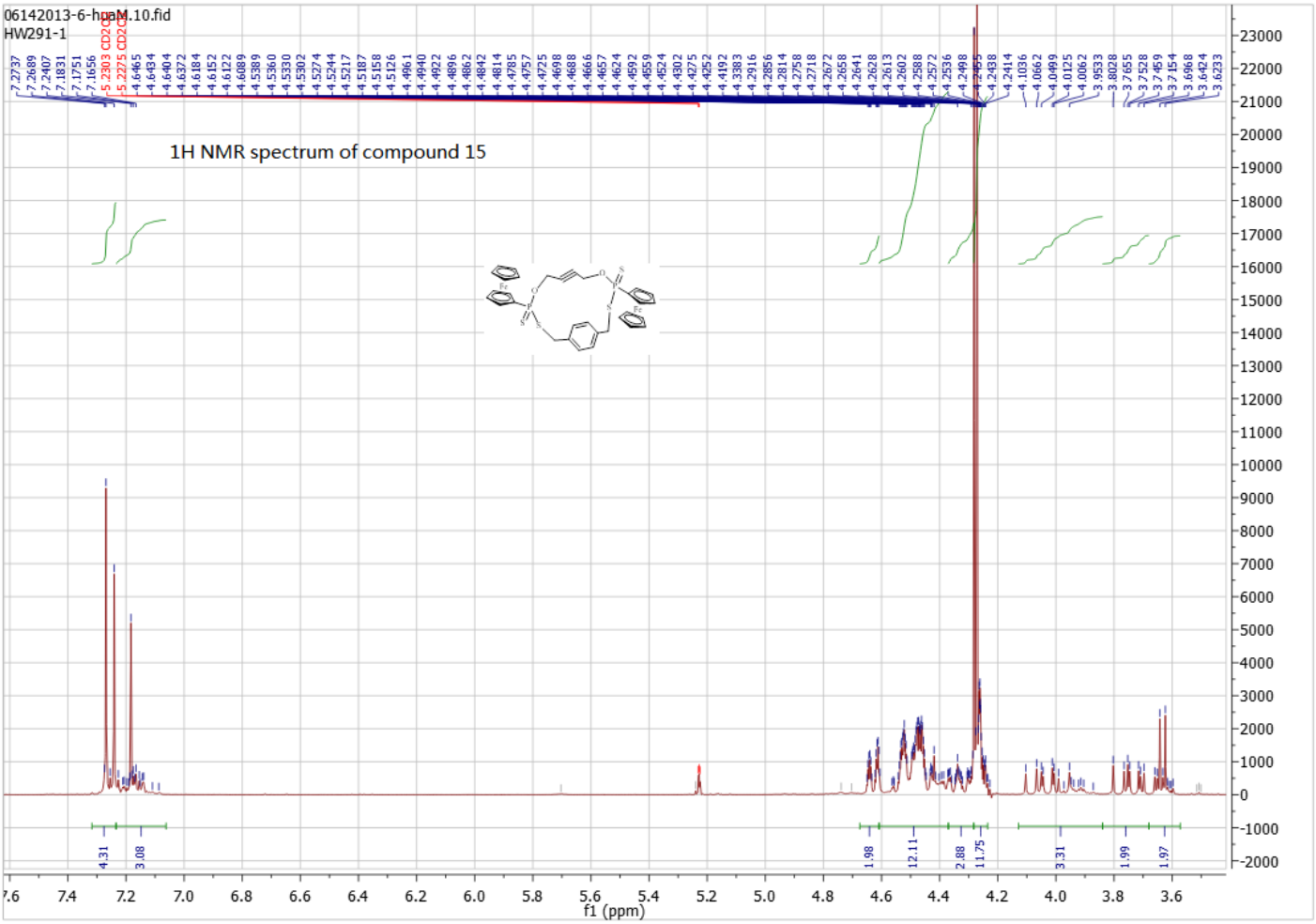

05212013-38-huaM.11.fid
HW283-1
13C NMR spectrum of compound 12
131.0596
130.8539
128.5540
128.5384
72.4845
72.3389
72.2486
72.2250
72.1175
72.0911
71.9947
71.8907
71.5088
71.3915
71.0979
70.9617
70.9216
70.8095
70.7453
70.5975
70.3747
70.3253
34.9219
13.9901
13.9575
13.8744
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
-6000
-8000
-10000
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

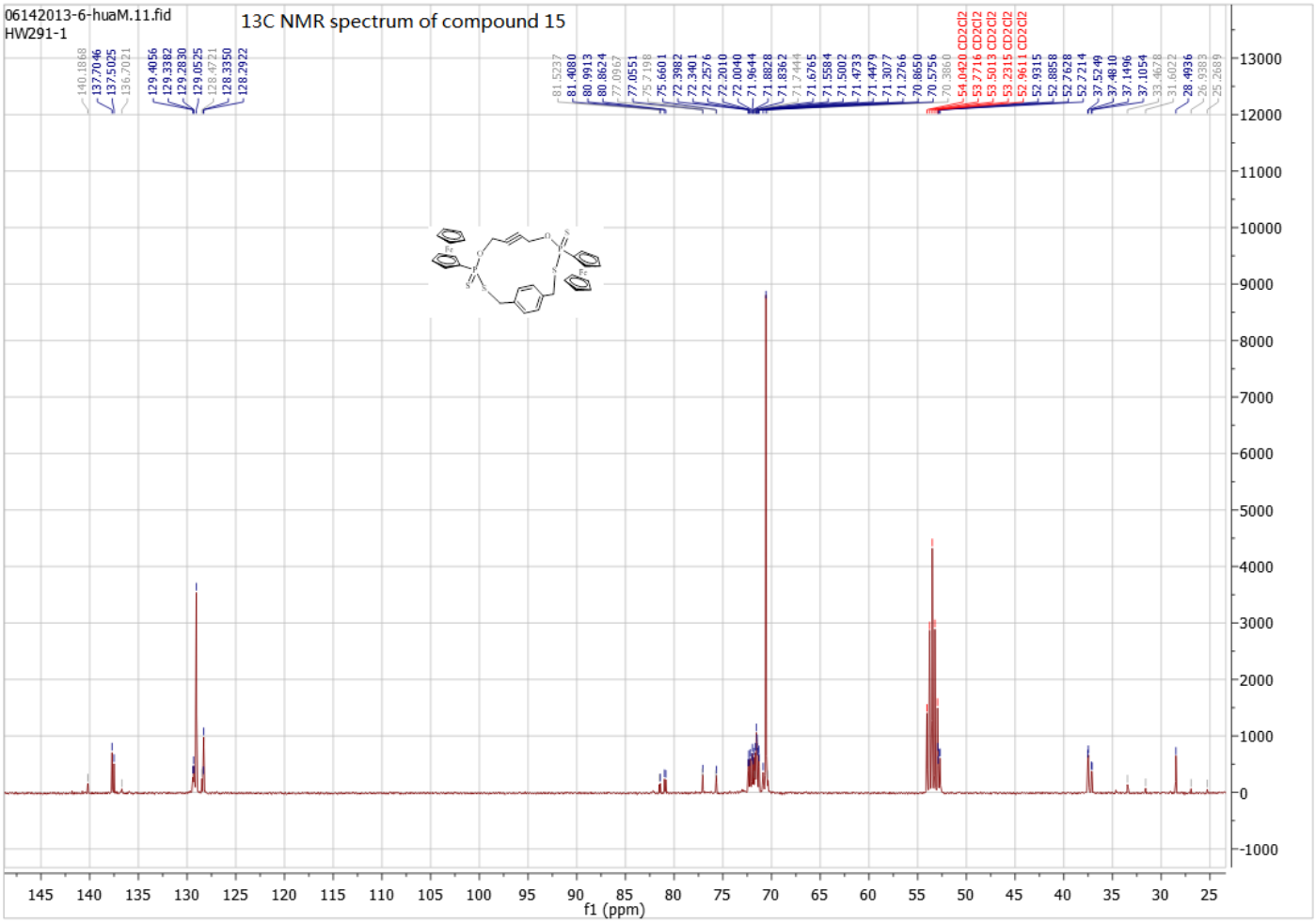

05222013-49-huaM.10.fid
HW285-1
1H NMR spectrum of compound 13
f1 (ppm)

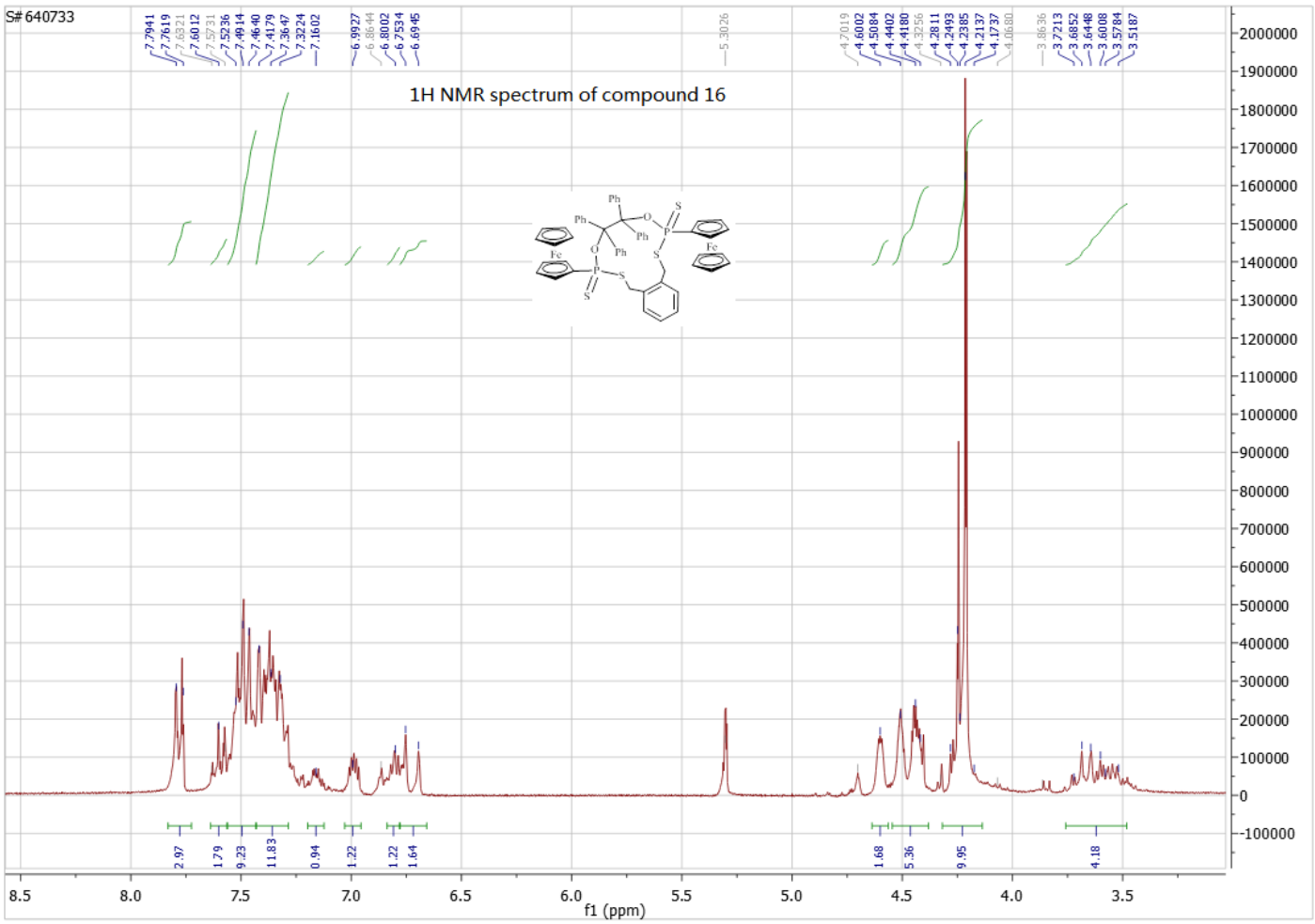

05222013-49-huaM.12.fid
HW285-1
13C NMR spectrum of compound 13
f1 (ppm)

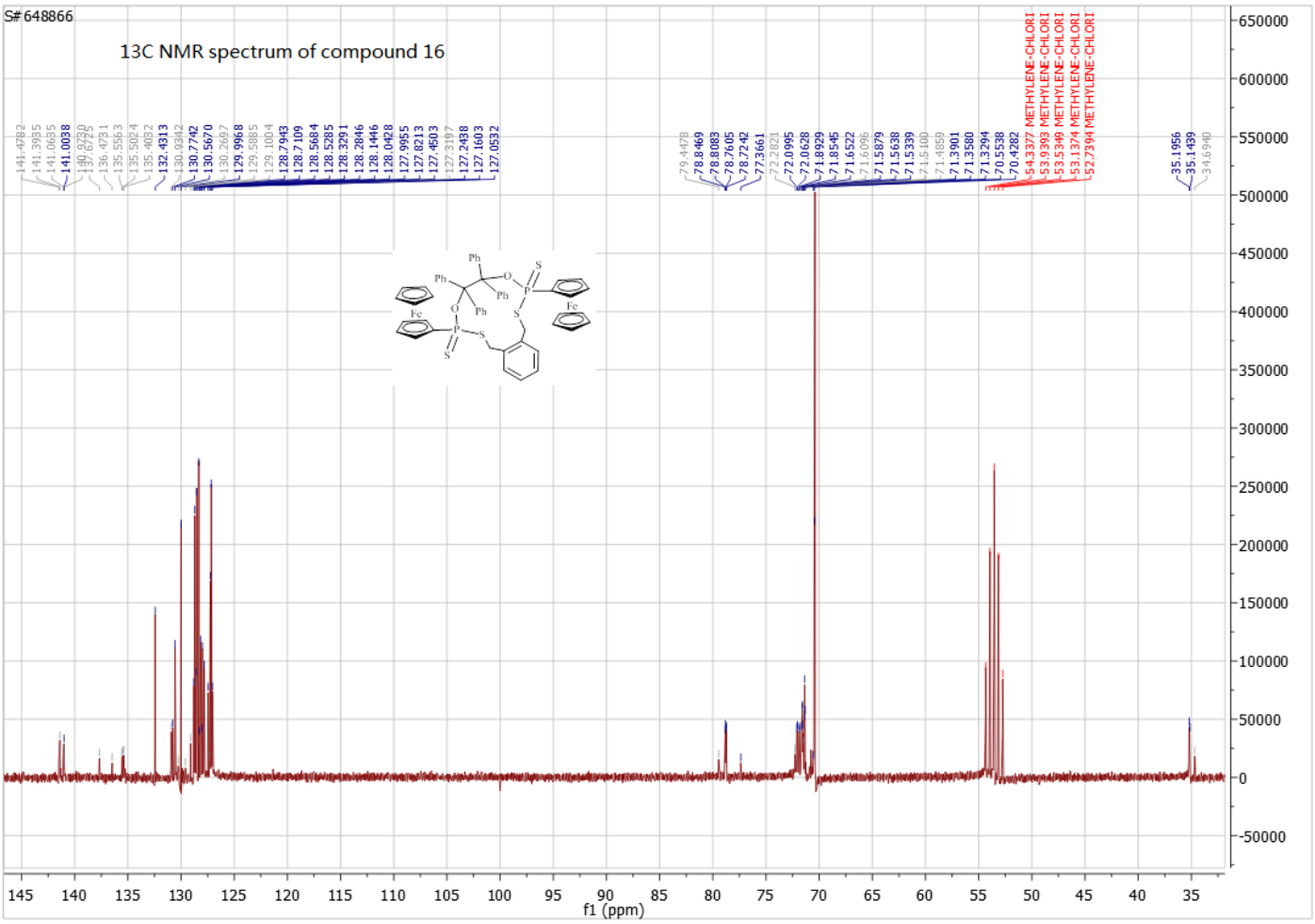

05242013-9-hpaM.10.fid
HW286-1
1H NMR spectrum of compound 14
f1 (ppm)

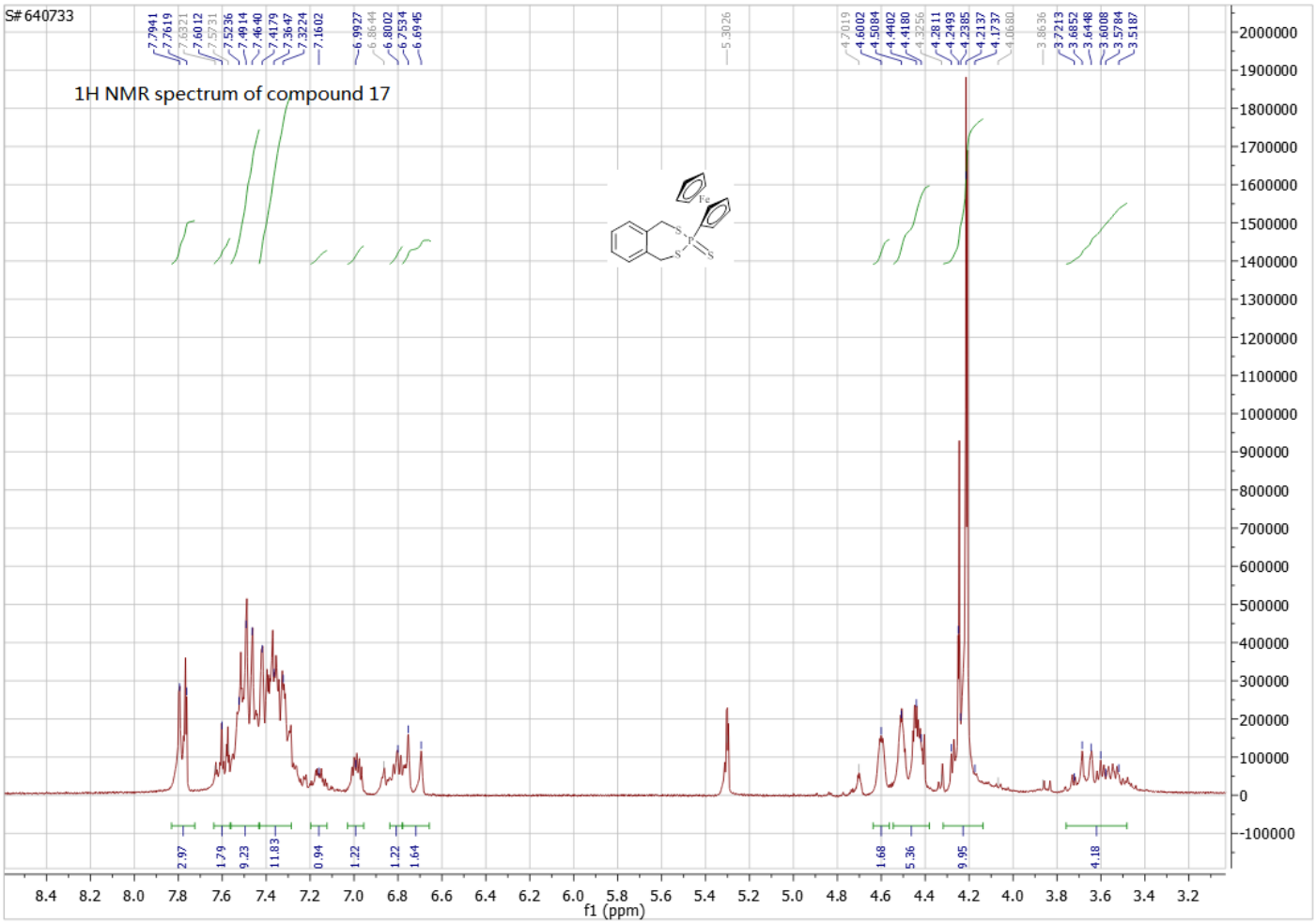

05242013-9-huaM.11.fid
HW286-1
13C NMR spectrum of compound 14
129.8588
129.7912
129.4030
129.3557
72.2488
72.1112
72.0734
71.9754
71.9419
71.8223
71.7019
71.2462
71.0969
71.0385
70.9779
70.9151
70.8209
70.6496
70.4338
70.3642
70.3312
14.0102
13.8671
f1 (ppm)

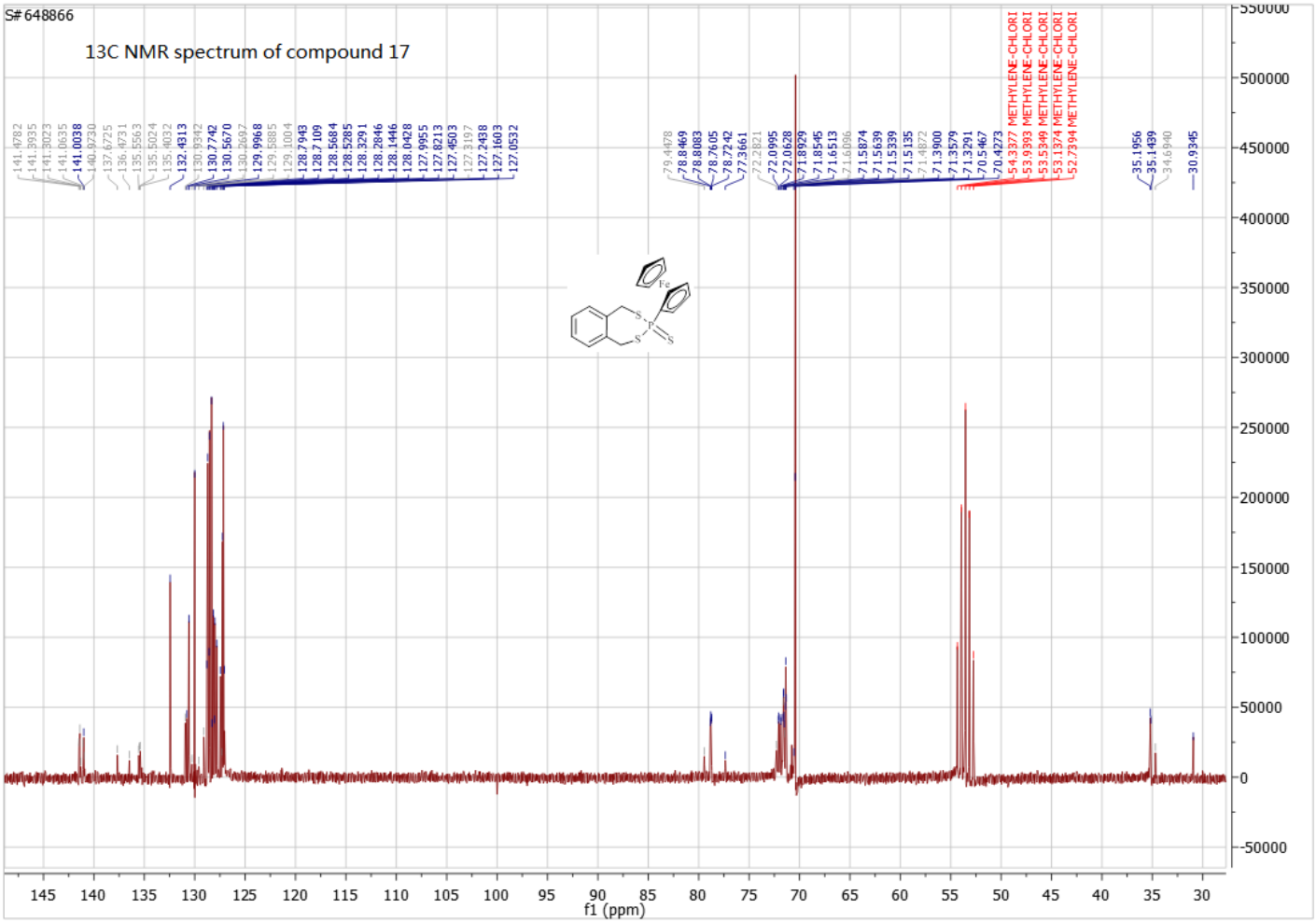

06142013-6-hpaM.10.fid
HW291-1
1H NMR spectrum of compound 15
f1 (ppm)

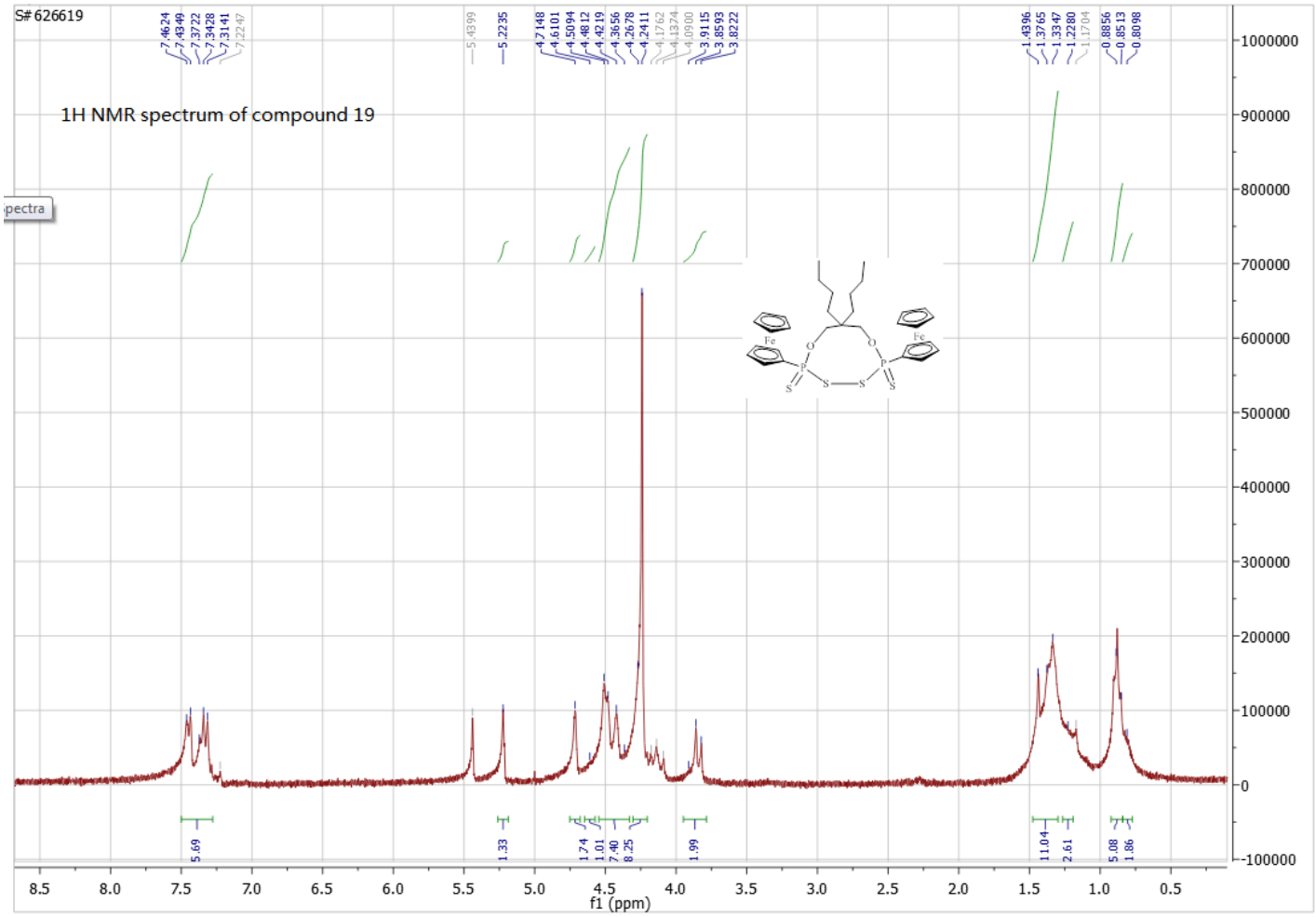

06142013-6-huaM.11.fid
HW291-1
13C NMR spectrum of compound 15
f1 (ppm)

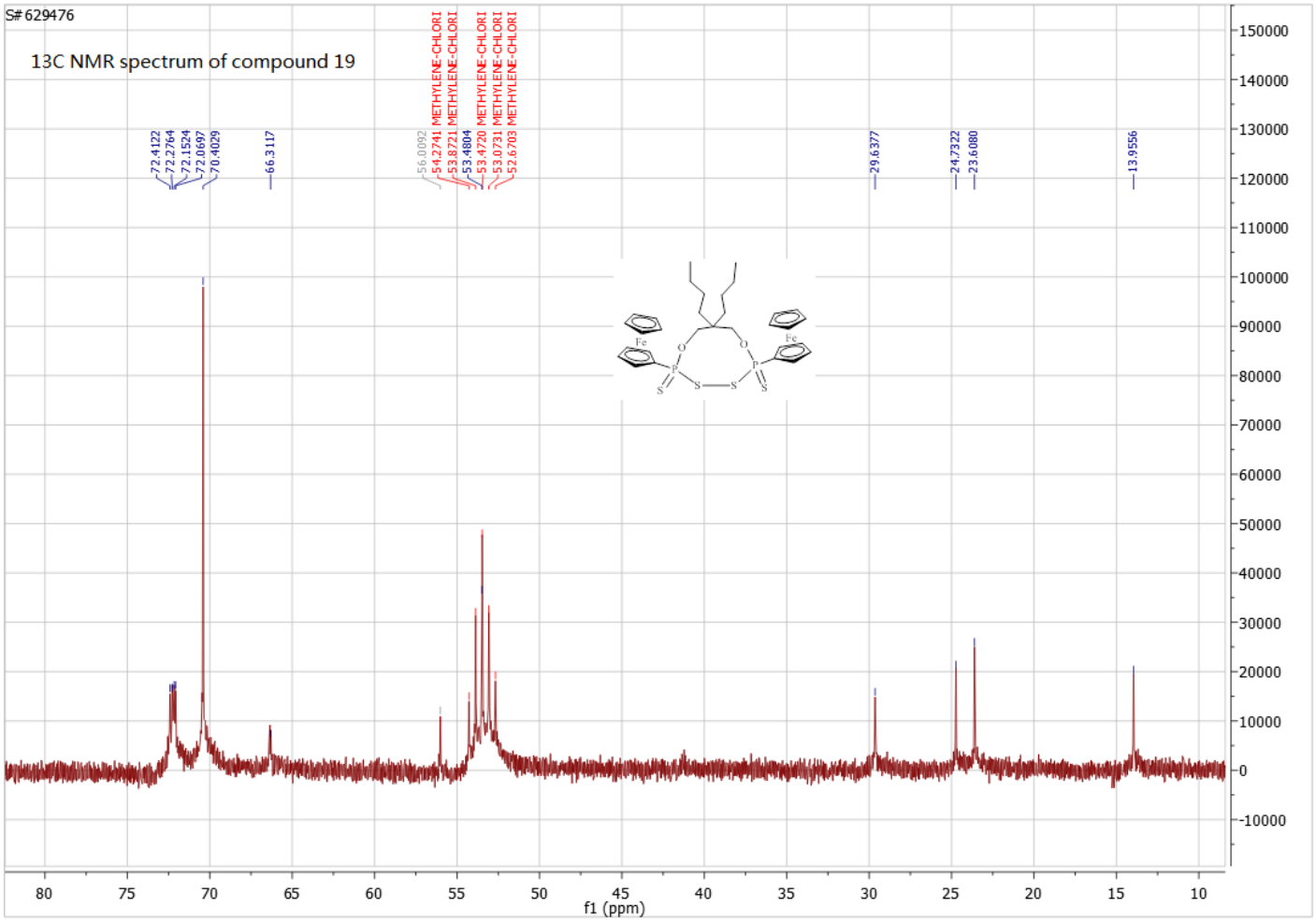

S# 640733
1H NMR spectrum of compound 16
f1 (ppm)

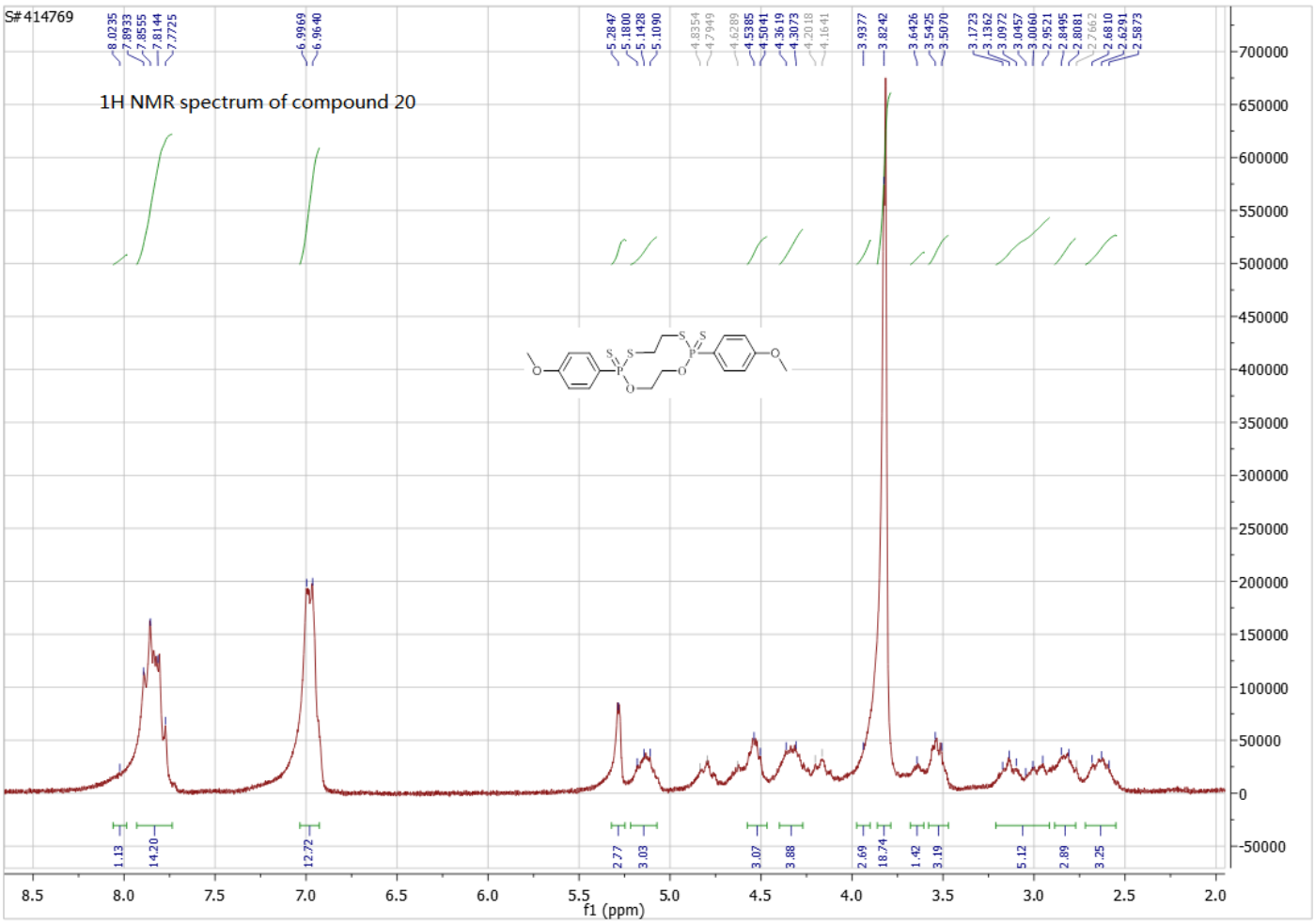

S#648866
13C NMR spectrum of compound 16
f1 (ppm)

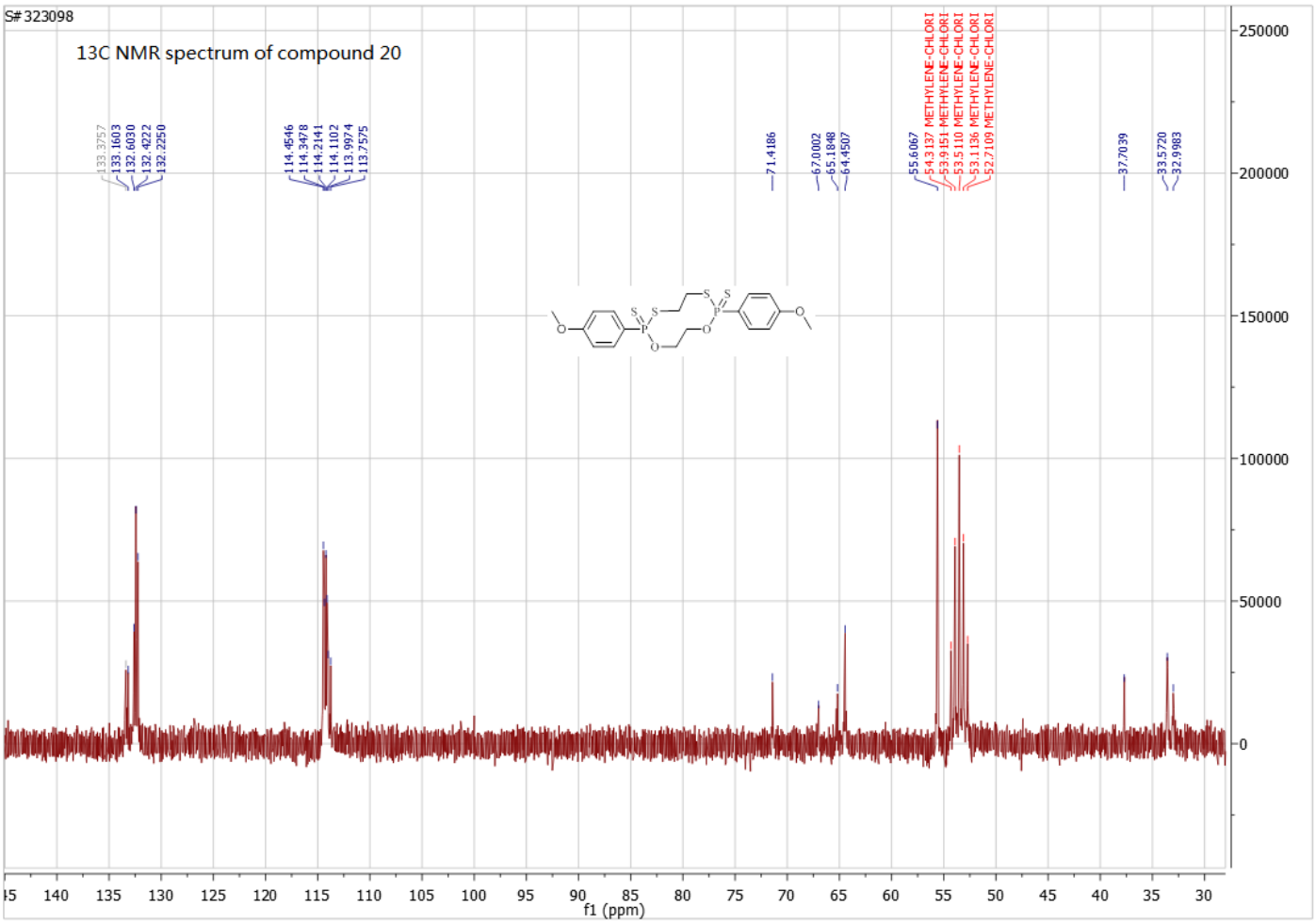

S# 640733
1H NMR spectrum of compound 17
f1 (ppm)

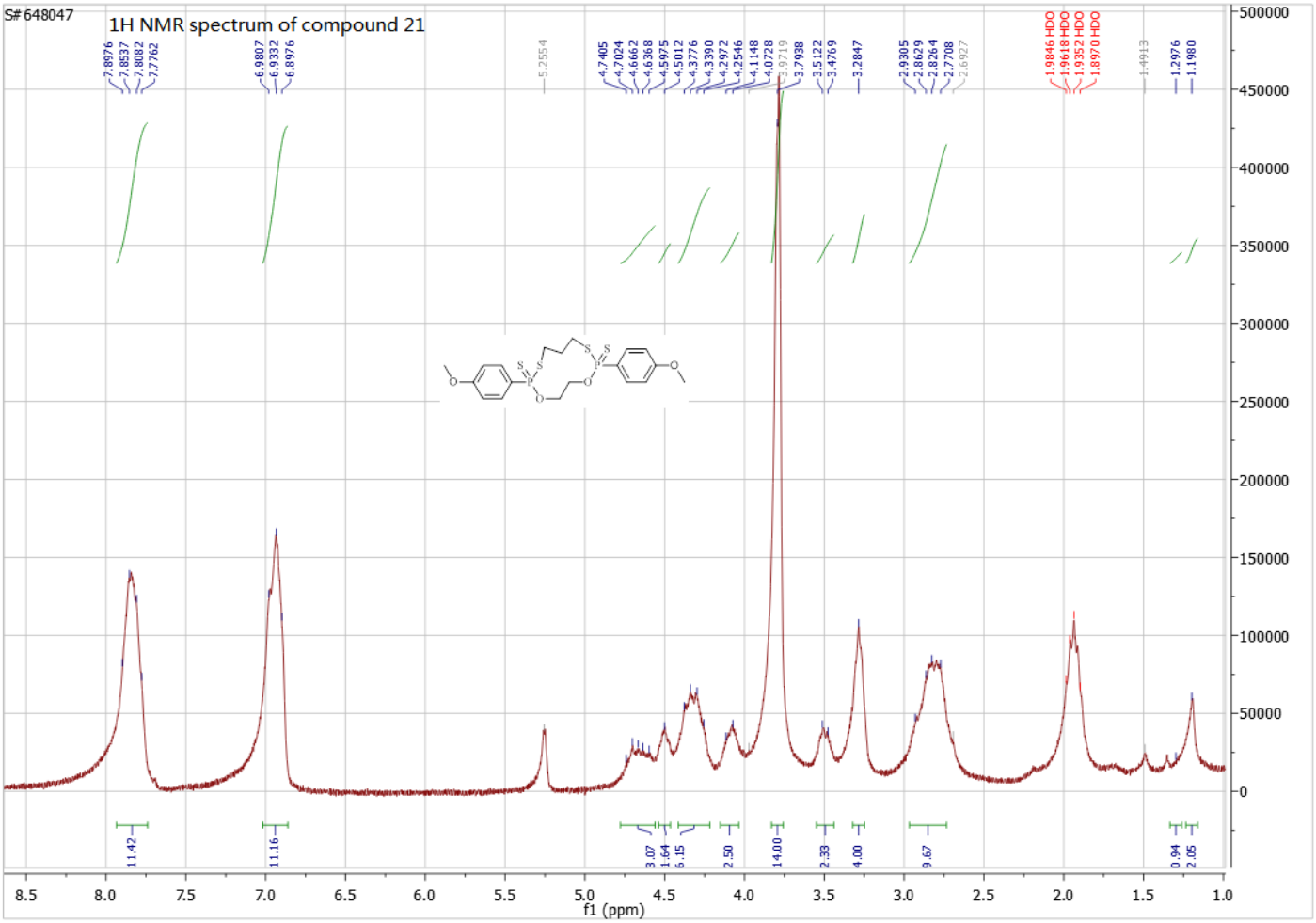

S# 648866
13C NMR spectrum of compound 17
f1 (ppm)

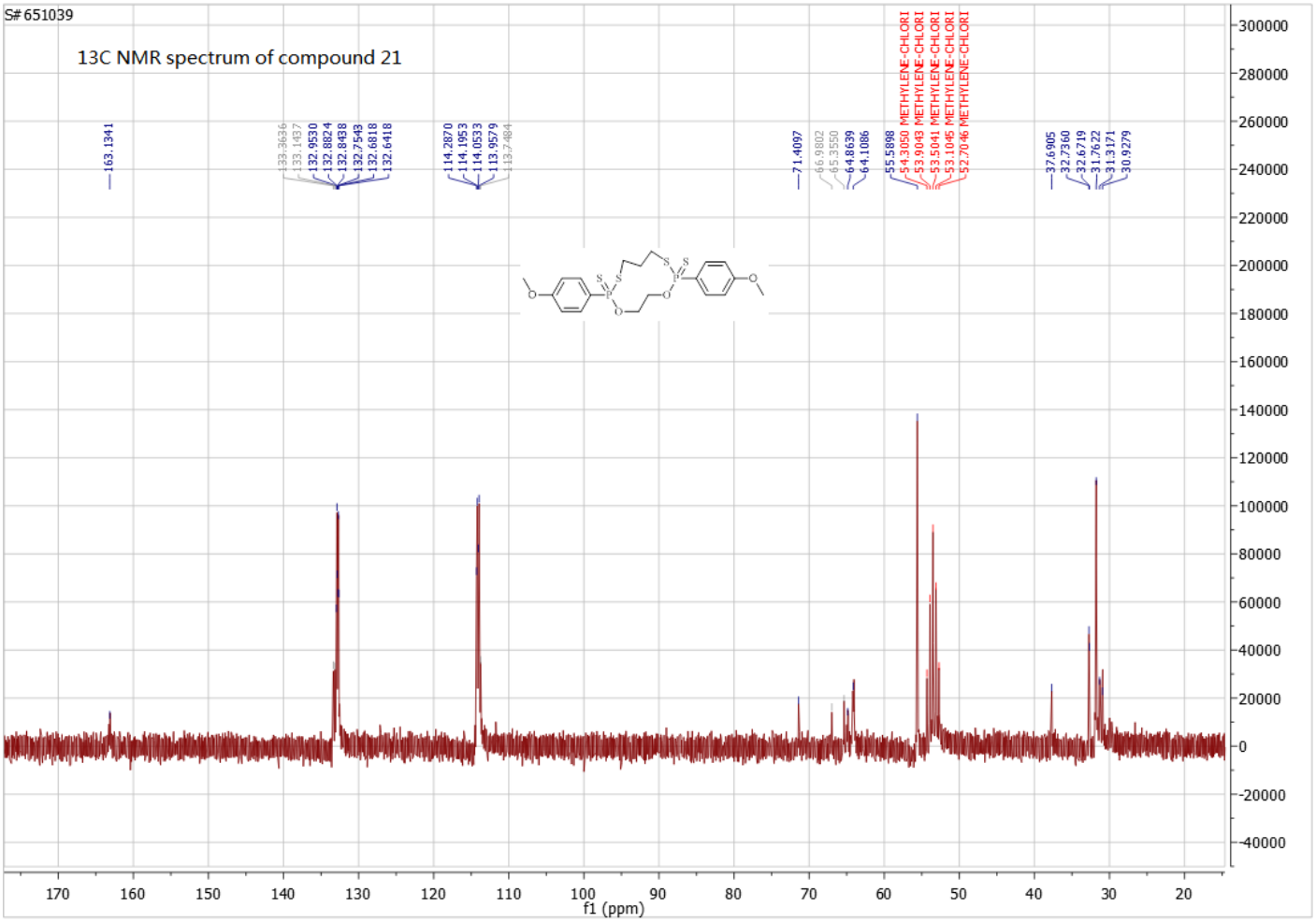

S# 626619
1H NMR spectrum of compound 19
7.4624
7.4349
7.3722
7.3428
7.3141
7.2247
5.4399
5.2235
4.7148
4.6101
4.5094
4.4812
4.4219
4.3656
4.2678
4.2411
4.1762
4.1374
4.0900
3.9115
3.8593
3.8222
1.4396
1.3765
1.3347
1.2280
1.1704
0.8856
0.8513
0.8098
5.69
1.33
1.74
1.01
7.40
8.25
1.99
11.04
2.61
5.08
1.86
f1 (ppm)

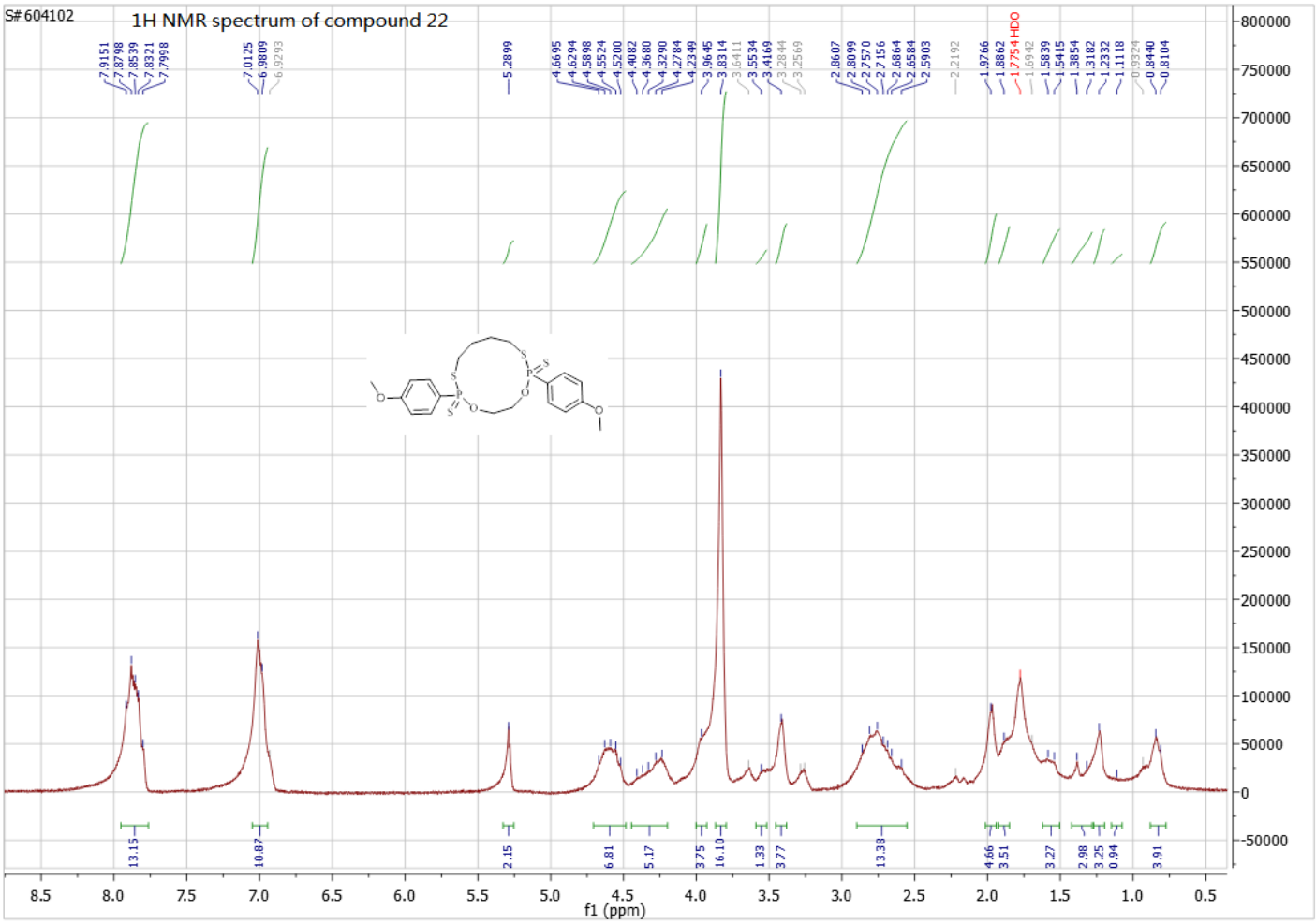

S# 629476
13C NMR spectrum of compound 19
72.4122
72.2764
72.1524
72.0697
70.4029
66.3117
56.0092
54.2741 METHYLENE-CHLORI
53.8721 METHYLENE-CHLORI
53.4804
53.4720 METHYLENE-CHLORI
53.0731 METHYLENE-CHLORI
52.6703 METHYLENE-CHLORI
29.6377
24.7322
23.6080
13.9556
Fe
Fe
O
O
P
P
S
S
S
S
150000
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
f1 (ppm)

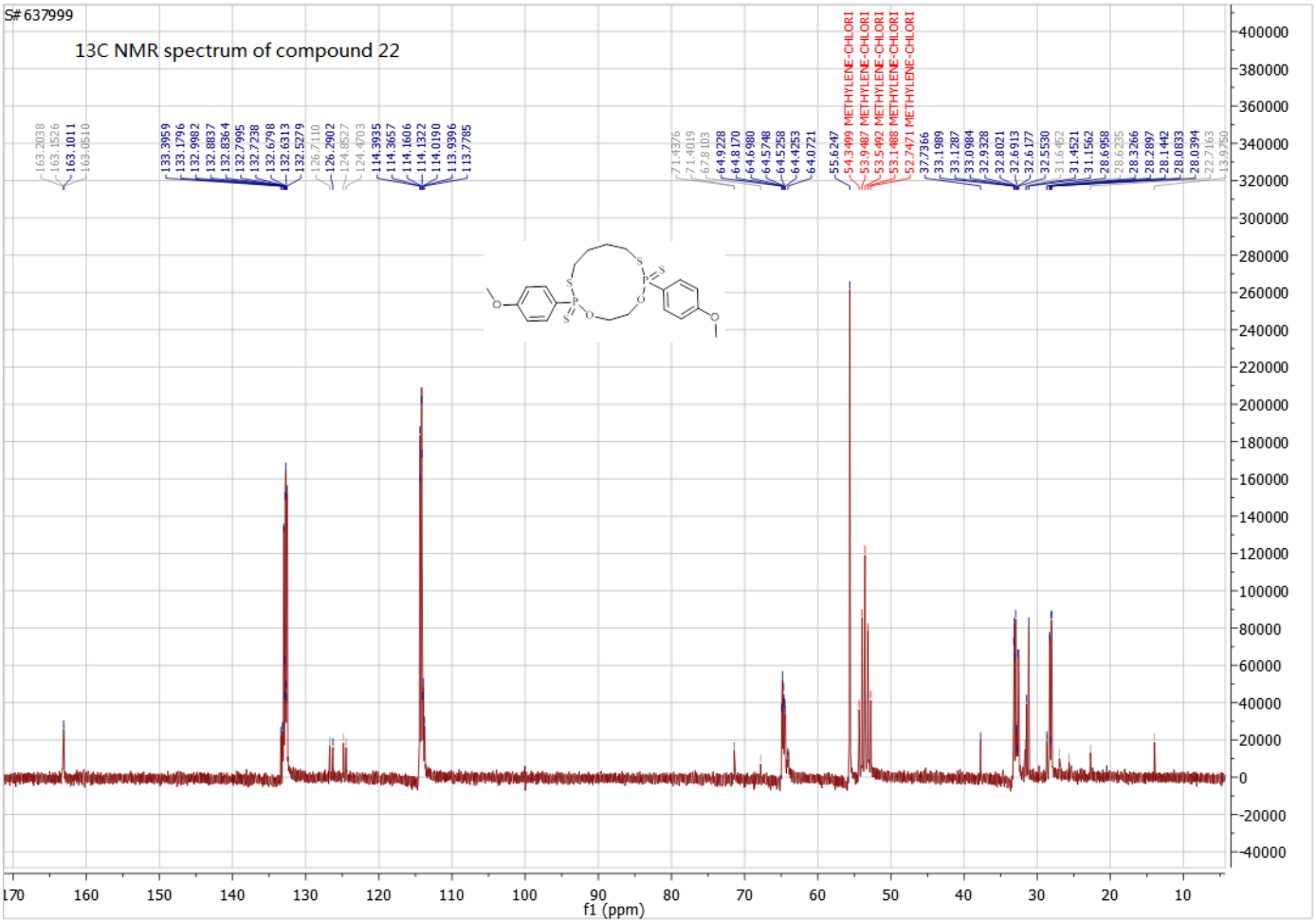

S#414769
1H NMR spectrum of compound 20
f1 (ppm)

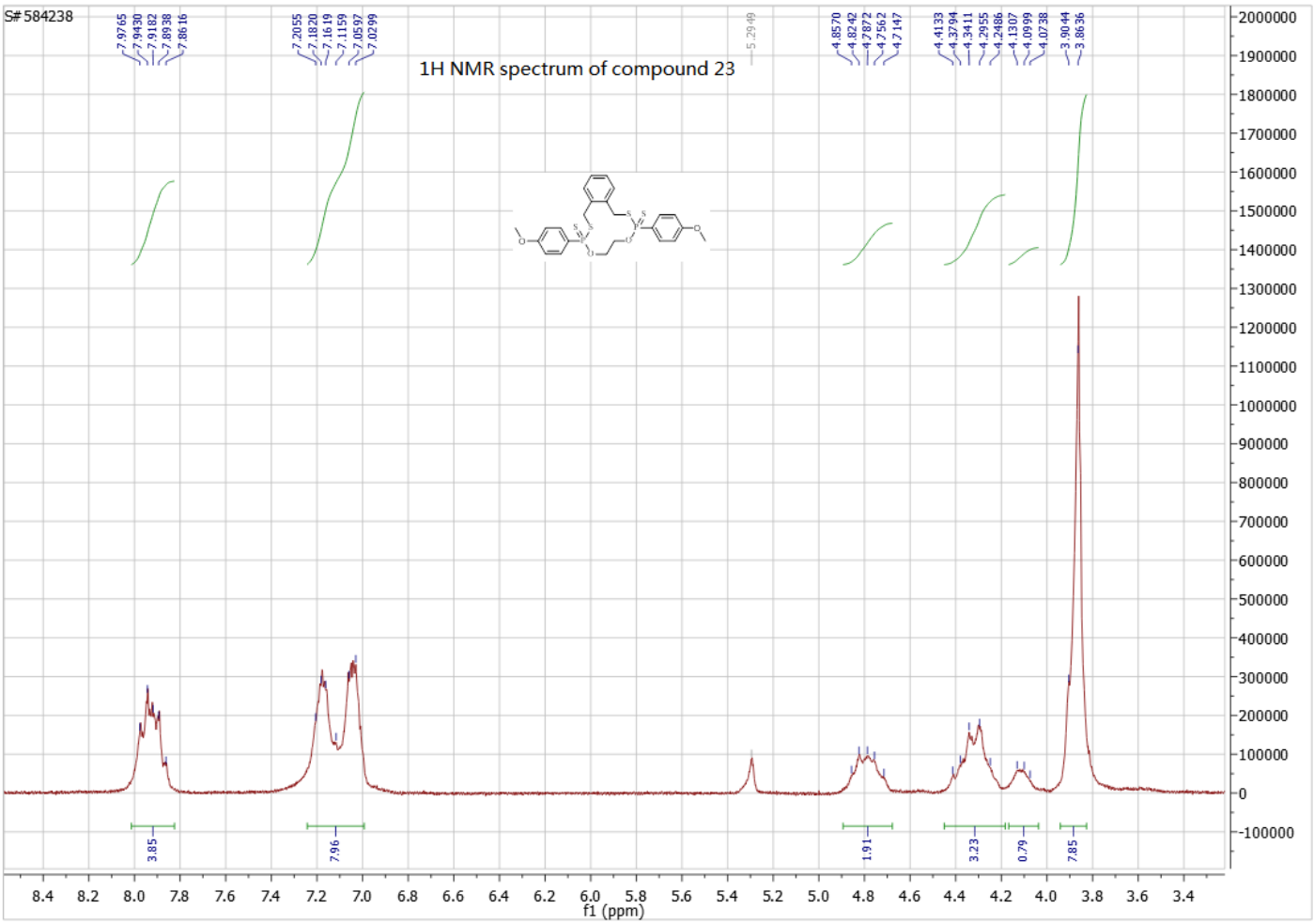

S# 323098
13C NMR spectrum of compound 20
133.3757
133.1603
132.6030
132.4222
132.2250
114.4546
114.3478
114.2141
114.1102
113.9974
113.7575
71.4186
67.0002
65.1848
64.4507
55.6067
54.3137 METHYLENE-CHLORI
53.9151 METHYLENE-CHLORI
53.5110 METHYLENE-CHLORI
53.1136 METHYLENE-CHLORI
52.7109 METHYLENE-CHLORI
37.7039
33.5720
32.9983
250000
200000
150000
100000
50000
0
140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30
f1 (ppm)

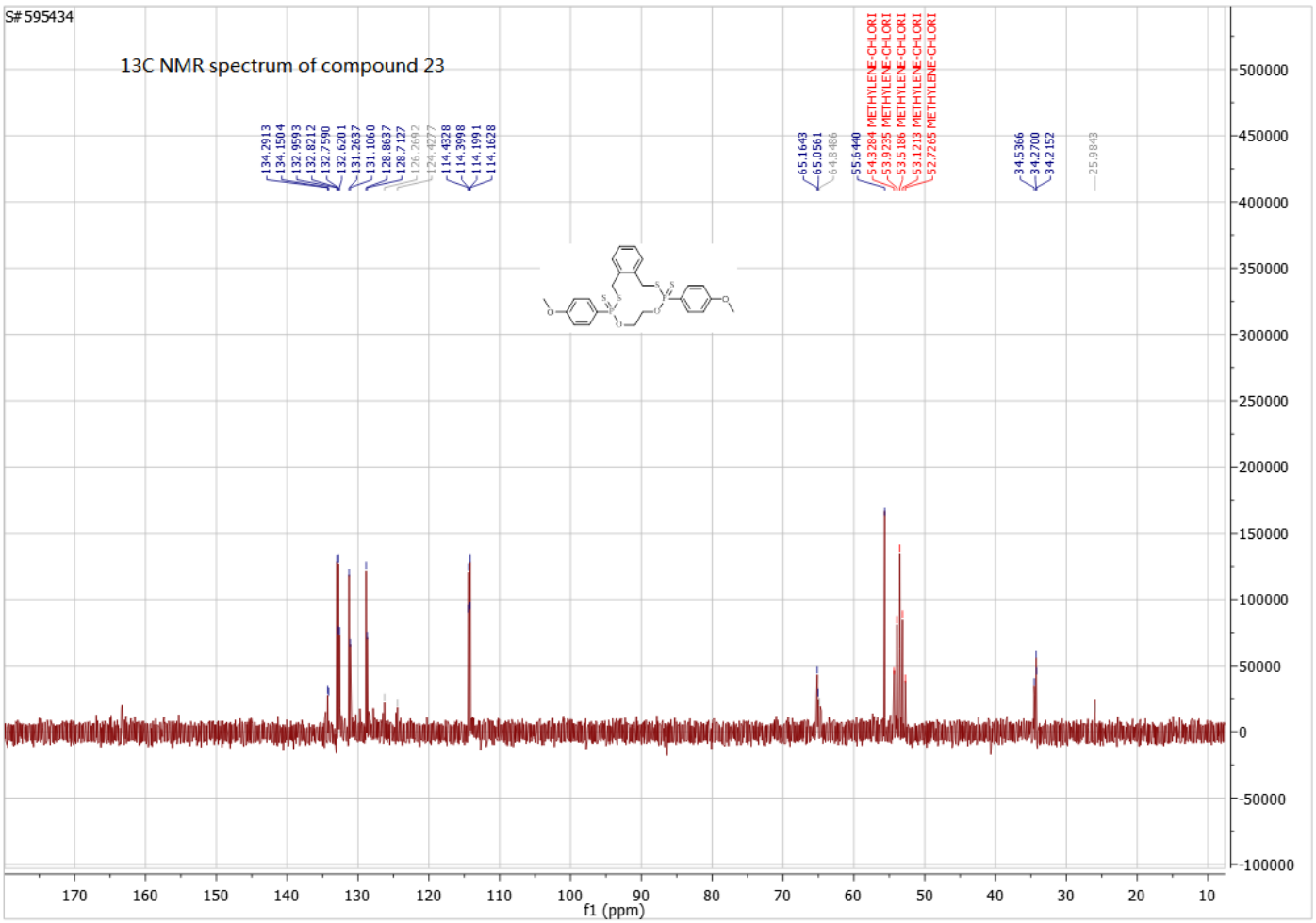

S# 648047
1H NMR spectrum of compound 21
7.8976
7.8537
7.8082
7.7762
6.9807
6.9332
6.8976
5.2554
4.7405
4.7024
4.6662
4.6368
4.5975
4.5012
4.3776
4.3390
4.2972
4.2546
4.1148
4.0728
3.9719
3.7938
3.5122
3.4769
3.2847
2.9305
2.8629
2.8264
2.7708
2.6927
1.9846 HDO
1.9618 HDO
1.9352 HDO
1.8970 HDO
1.4913
1.2976
1.1980
11.42
11.16
3.07
1.64
6.15
2.50
14.00
2.33
4.00
9.67
0.94
2.05
f1 (ppm)


S# 651039
13C NMR spectrum of compound 21
163.1341
133.3636
133.1437
132.9530
132.8824
132.8438
132.7543
132.6818
132.6418
114.2870
114.1953
114.0533
113.9579
113.7484
71.4097
66.9802
65.3550
64.8639
64.1086
55.5898
54.3050 METHYLENE-CHLORI
53.9043 METHYLENE-CHLORI
53.5041 METHYLENE-CHLORI
53.1045 METHYLENE-CHLORI
52.7046 METHYLENE-CHLORI
37.6905
32.7360
32.6719
31.7622
31.3171
30.9279
300000
280000
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
-40000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


S#604102
1H NMR spectrum of compound 22
f1 (ppm)


S# 637999
13C NMR spectrum of compound 22
f1 (ppm)


S# 584238
1H NMR spectrum of compound 23
7.9765
7.9430
7.9182
7.8938
7.8616
7.2055
7.1820
7.1619
7.1159
7.0597
7.0299
5.2949
4.8570
4.8242
4.7872
4.7562
4.7147
4.4133
4.3794
4.3411
4.2955
4.2486
4.1307
4.0999
4.0738
3.9044
3.8636
3.85
7.96
1.91
3.23
0.79
7.85
f1 (ppm)


S# 595434
13C NMR spectrum of compound 23
134.2913
134.1504
132.9593
132.8212
132.7590
132.6201
131.2637
131.1060
128.8637
128.7127
126.2692
124.4227
114.4328
114.3998
114.1991
114.1628
65.1643
65.0561
64.8486
55.6440
54.3284 METHYLENE-CHLORI
53.9235 METHYLENE-CHLORI
53.5186 METHYLENE-CHLORI
53.1213 METHYLENE-CHLORI
52.7265 METHYLENE-CHLORI
34.5366
34.2700
34.2152
25.9843
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
-100000
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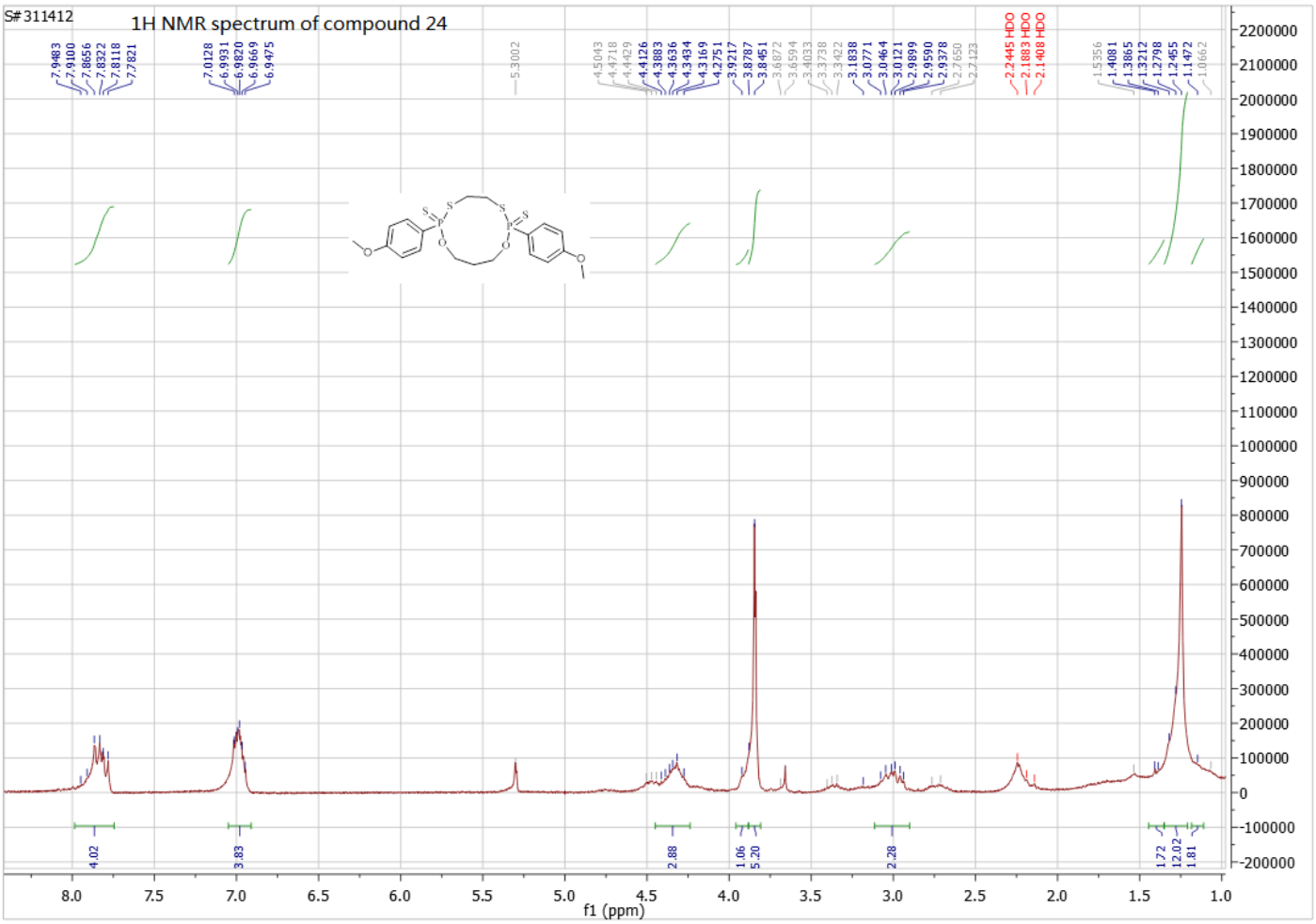
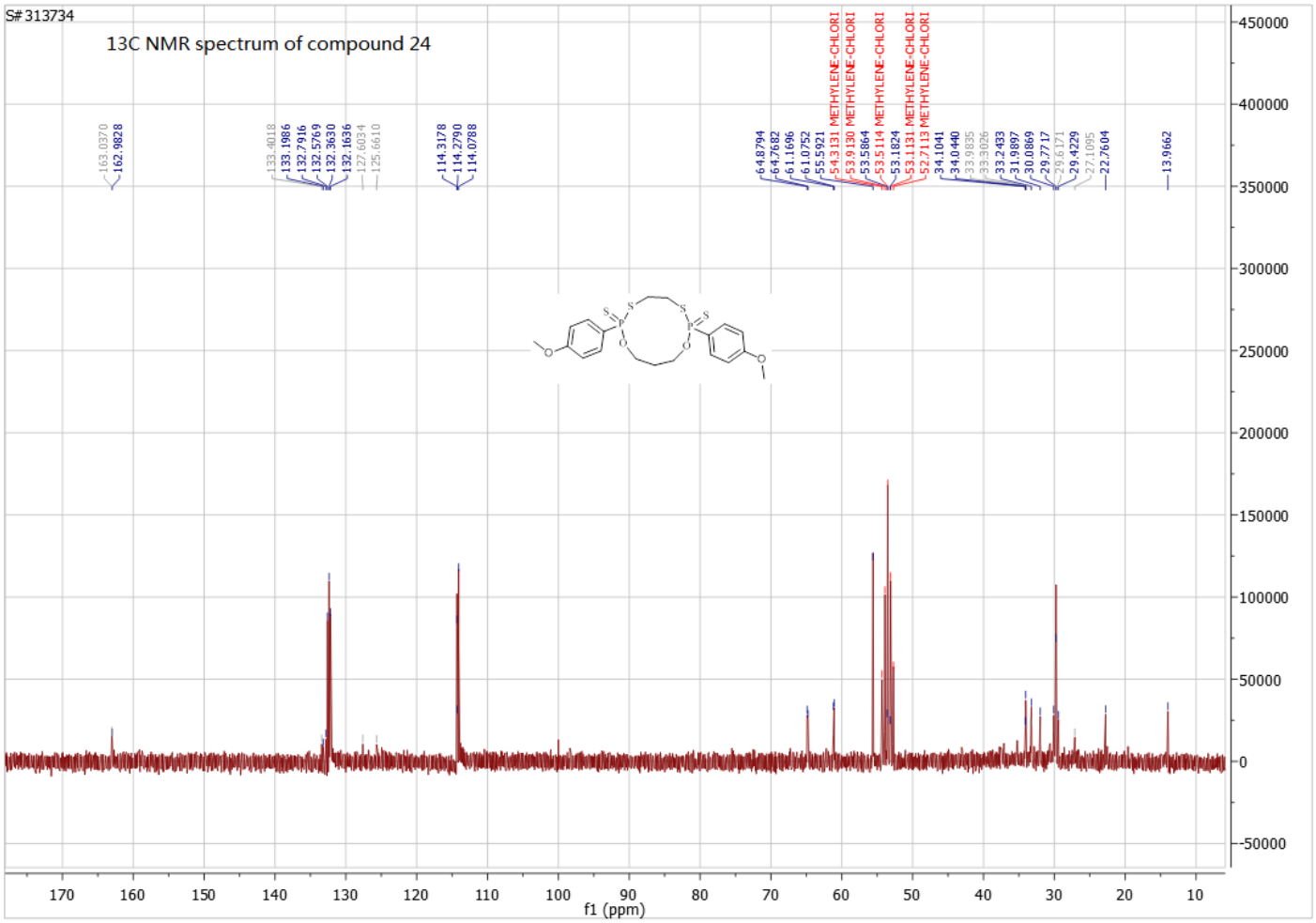
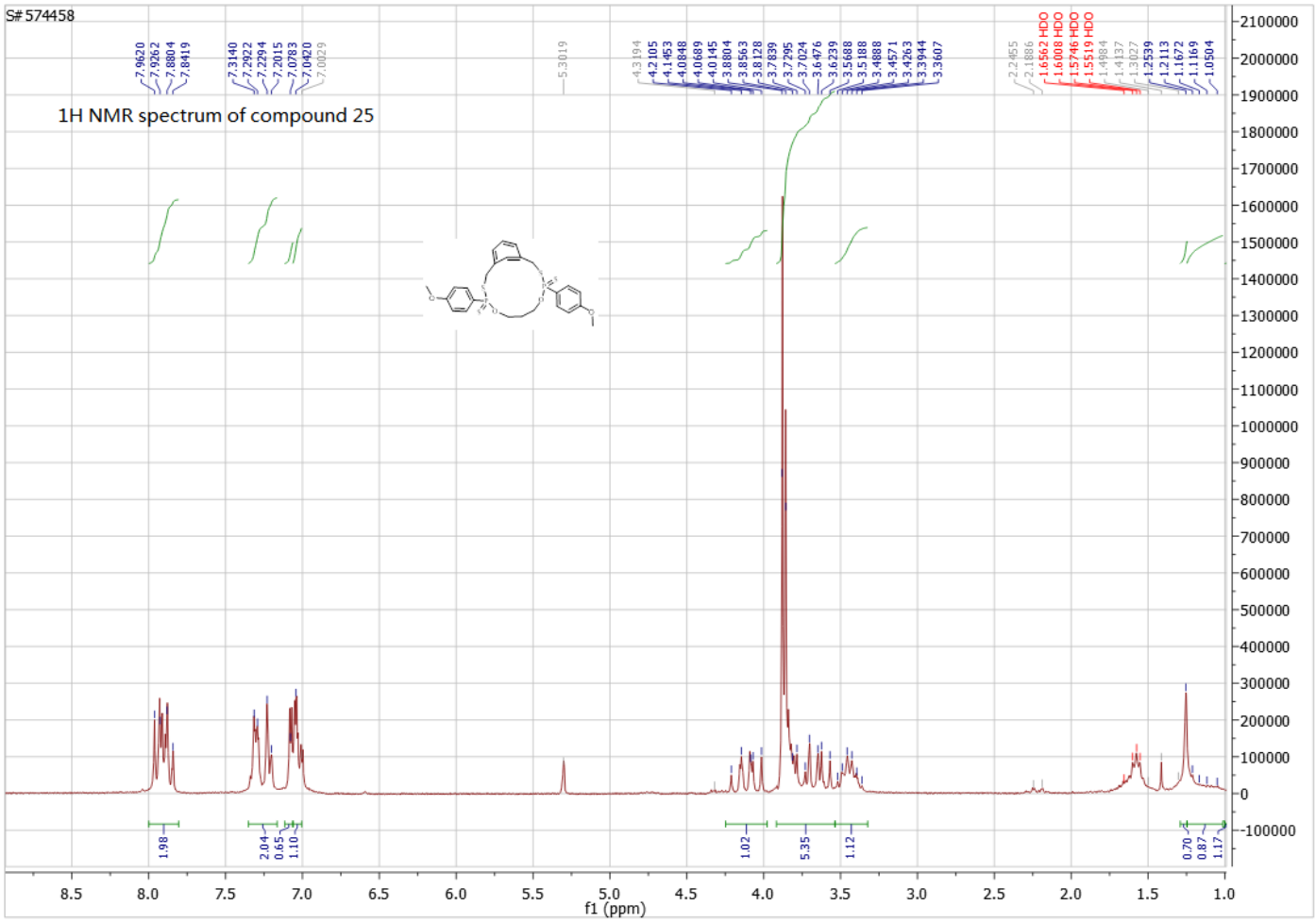
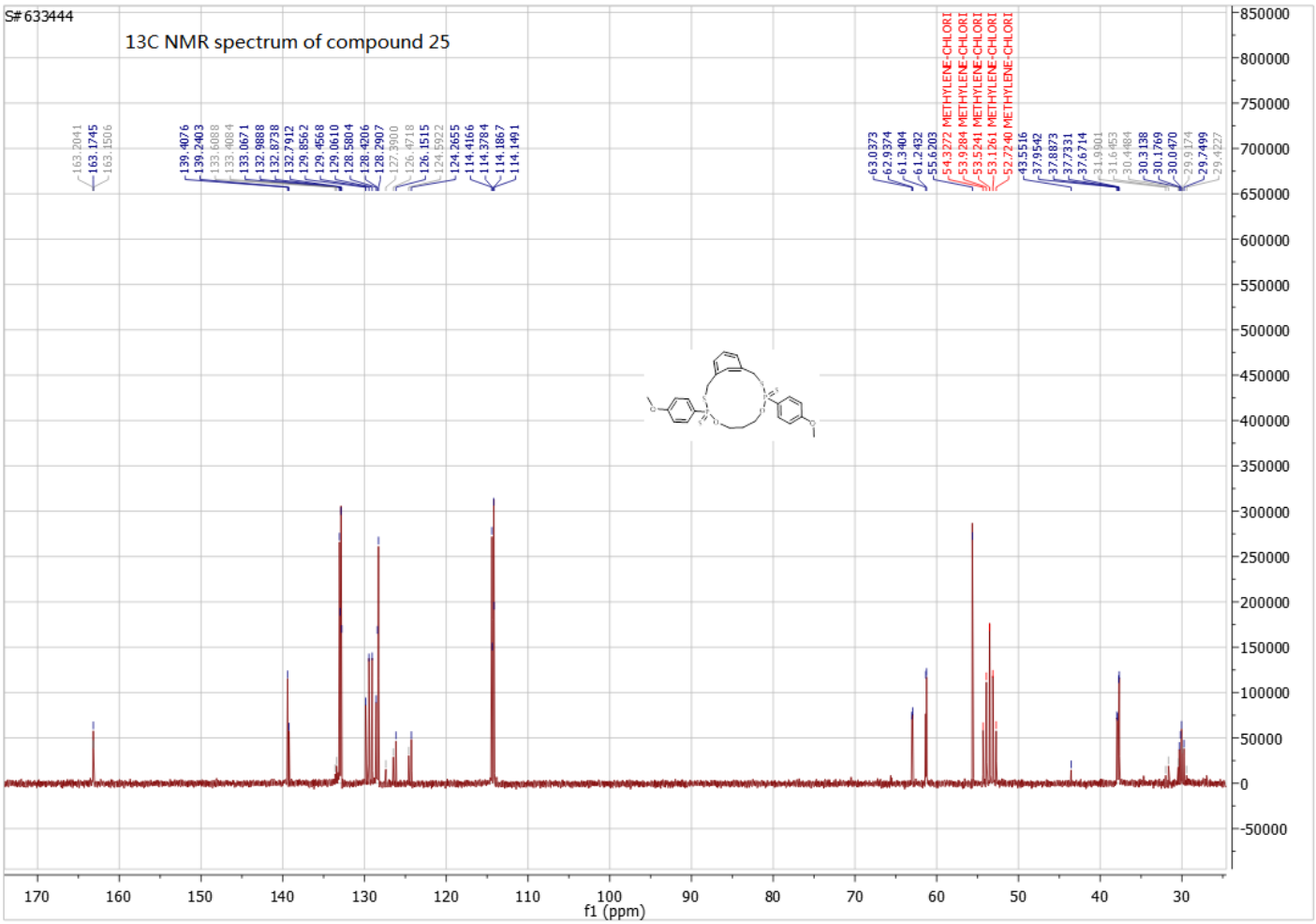
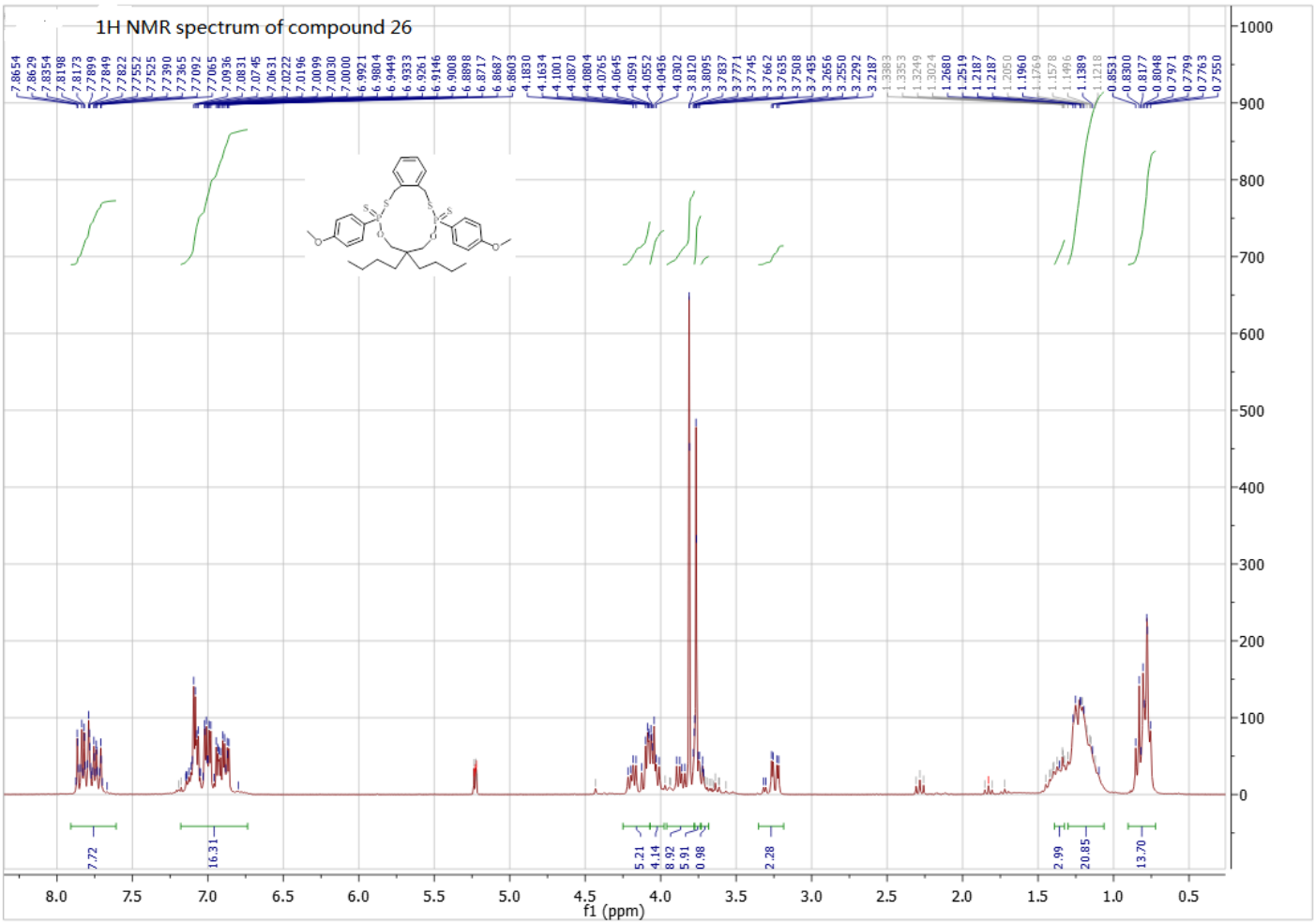
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

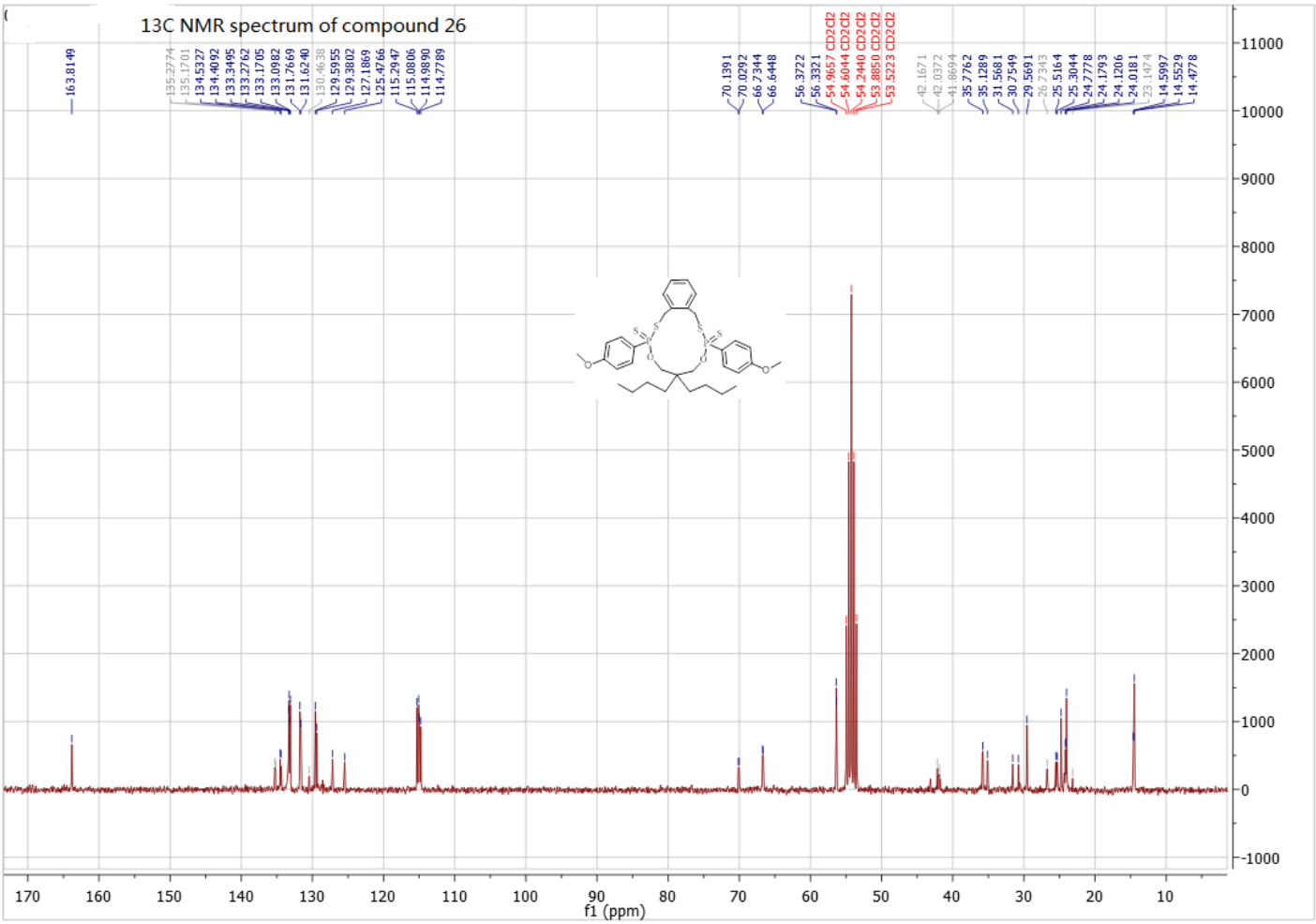

S#311412
1H NMR spectrum of compound 24
f1 (ppm)

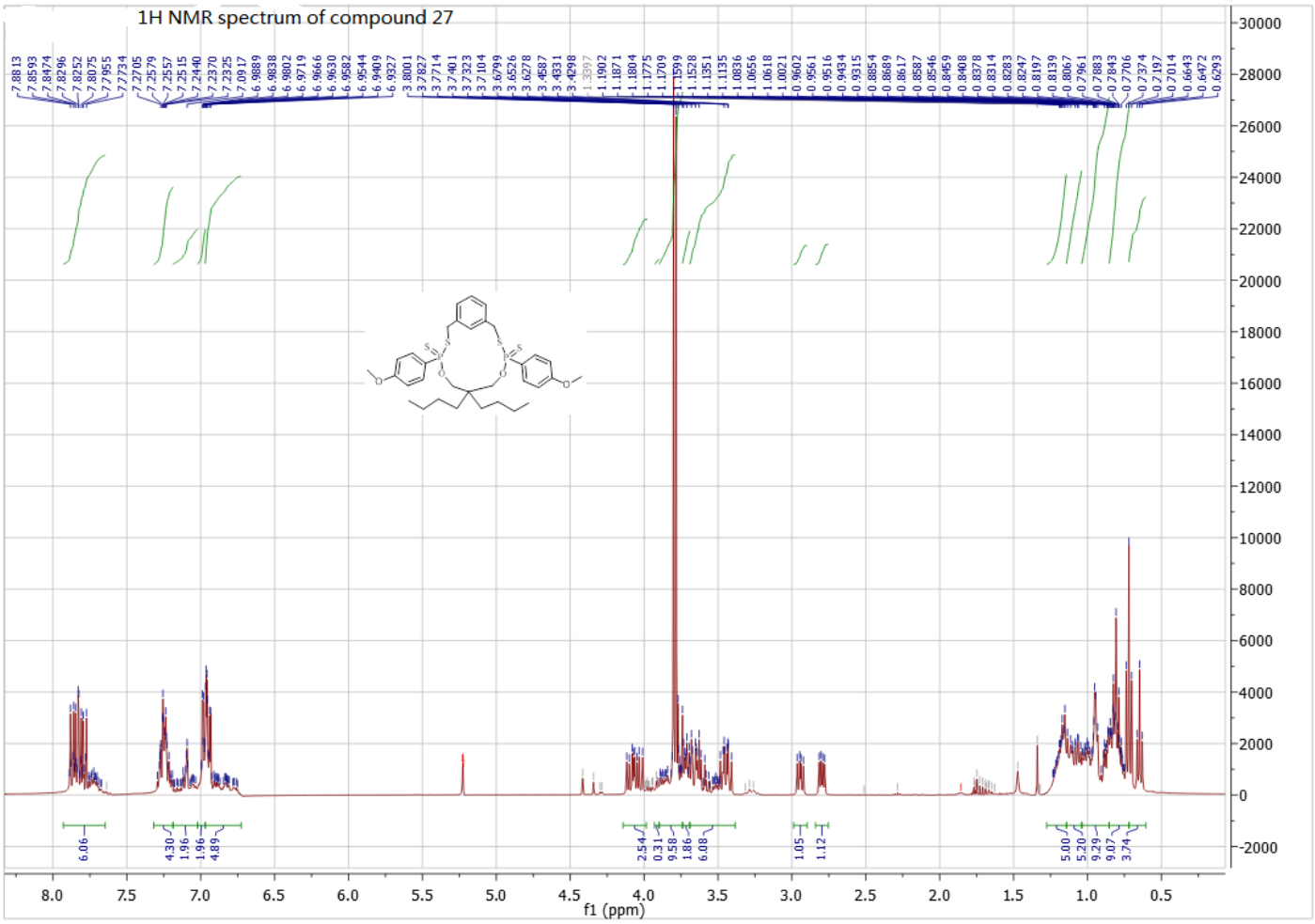

S#313734
13C NMR spectrum of compound 24
163.0370
162.9828
133.4018
133.1986
132.7916
132.5769
132.3630
132.1636
127.6034
125.6610
114.3178
114.2790
114.0788
64.8794
64.7682
61.1696
61.0752
55.5921
54.3131 METHYLENE-CHLORI
53.9130 METHYLENE-CHLORI
53.5864
53.5114 METHYLENE-CHLORI
53.1824
53.1131 METHYLENE-CHLORI
52.7113 METHYLENE-CHLORI
34.1041
34.0440
33.9835
33.9026
33.2433
31.9897
30.0869
29.7717
29.6171
29.4229
27.1095
22.7604
13.9662
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

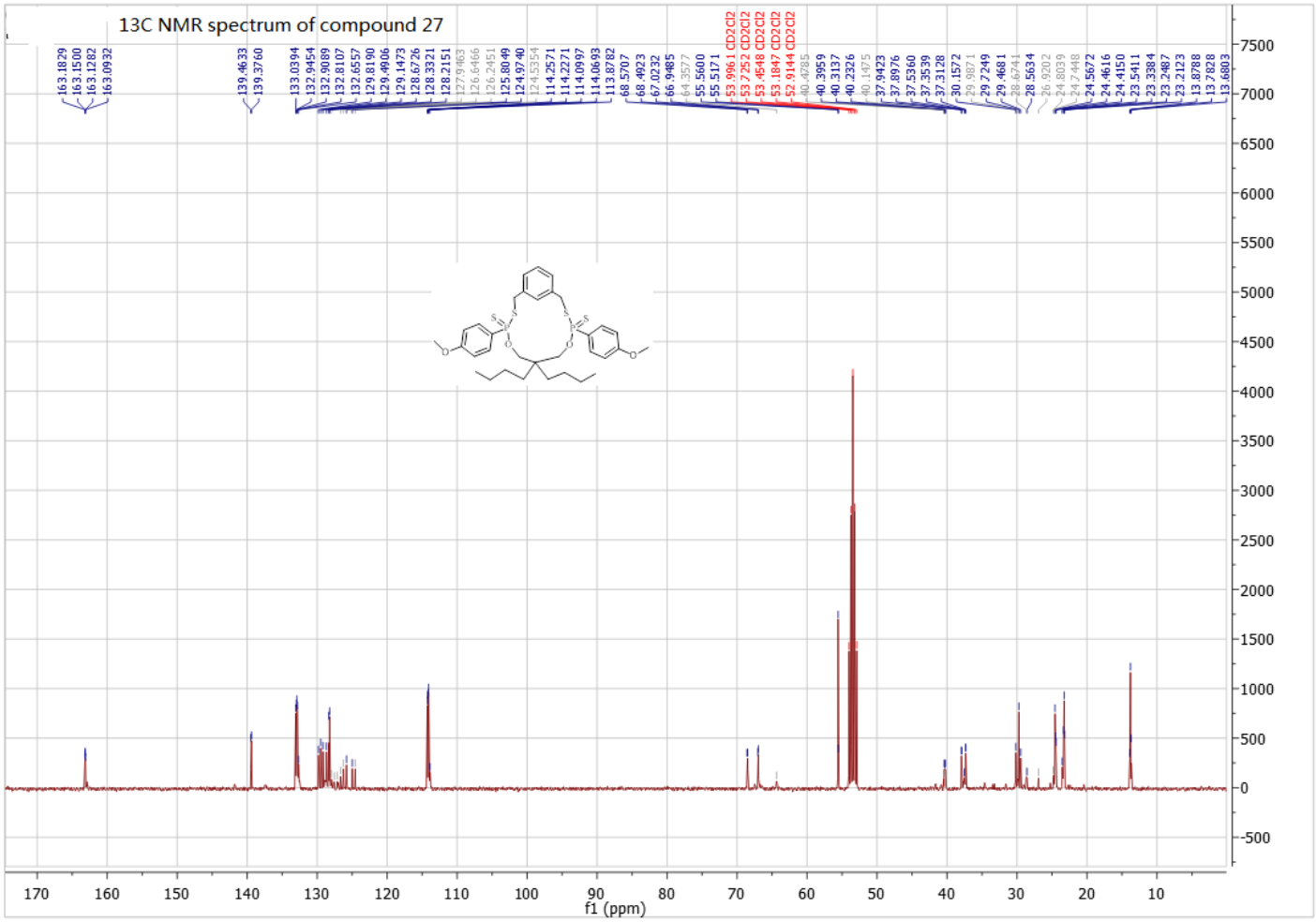

S# 574458
1H NMR spectrum of compound 25
f1 (ppm)

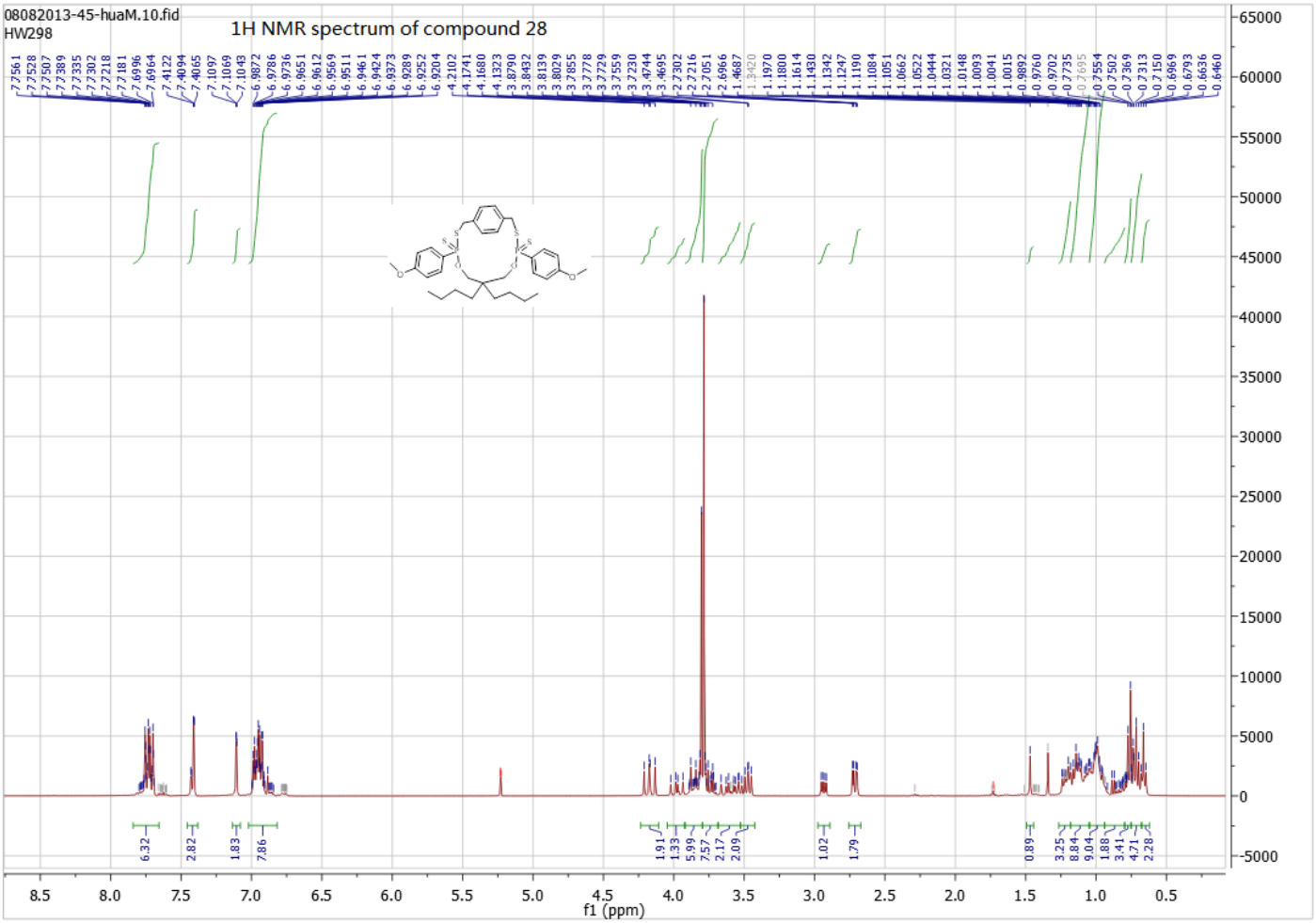

S# 633444
13C NMR spectrum of compound 25
f1 (ppm)

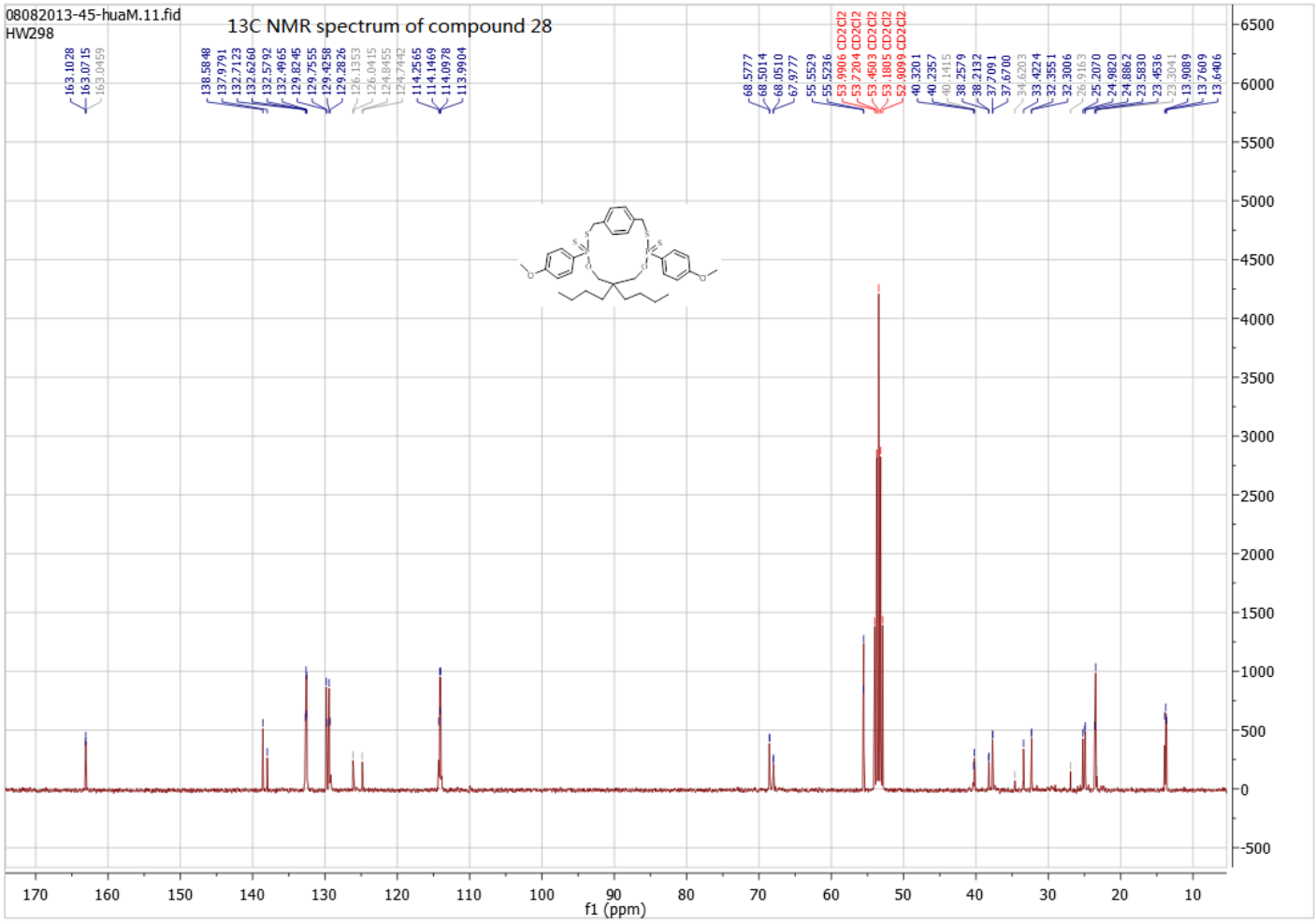

1H NMR spectrum of compound 26
f1 (ppm)

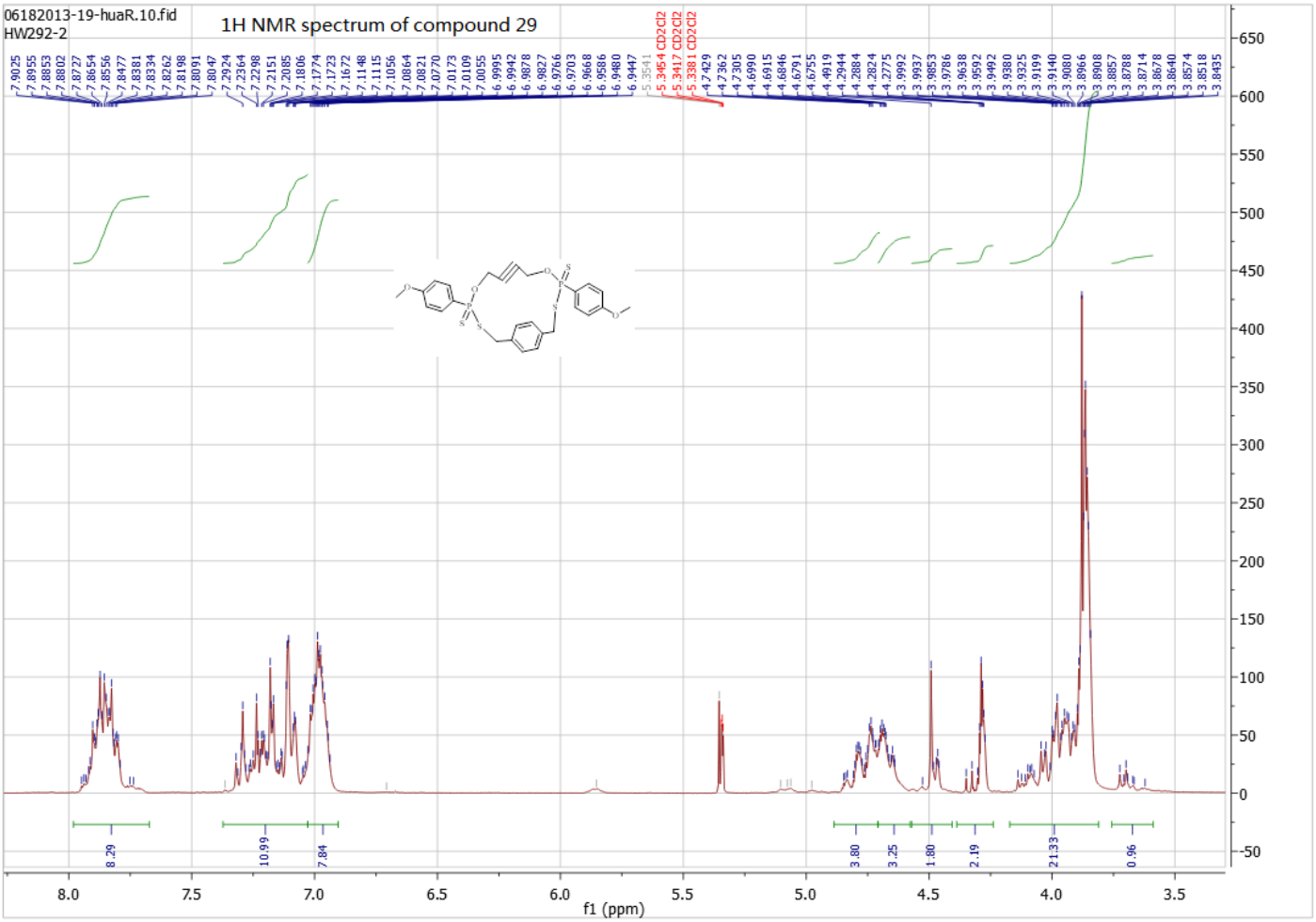

13C NMR spectrum of compound 26
163.8149
135.2774
135.1701
134.5327
134.4092
133.3495
133.2762
133.1705
133.0982
131.7669
131.6240
130.4638
129.5955
129.3802
127.1869
125.4766
115.2947
115.0806
114.9890
114.7789
70.1391
70.0292
66.7344
66.6448
56.3722
56.3321
54.9657 CD2Cl2
54.6044 CD2Cl2
54.2440 CD2Cl2
53.8850 CD2Cl2
53.5223 CD2Cl2
42.1671
42.0372
41.8694
35.7762
35.1289
31.5681
30.7549
29.5691
26.7343
25.5164
25.3044
24.7778
24.1793
24.1206
24.0181
23.1474
14.5997
14.5529
14.4778
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

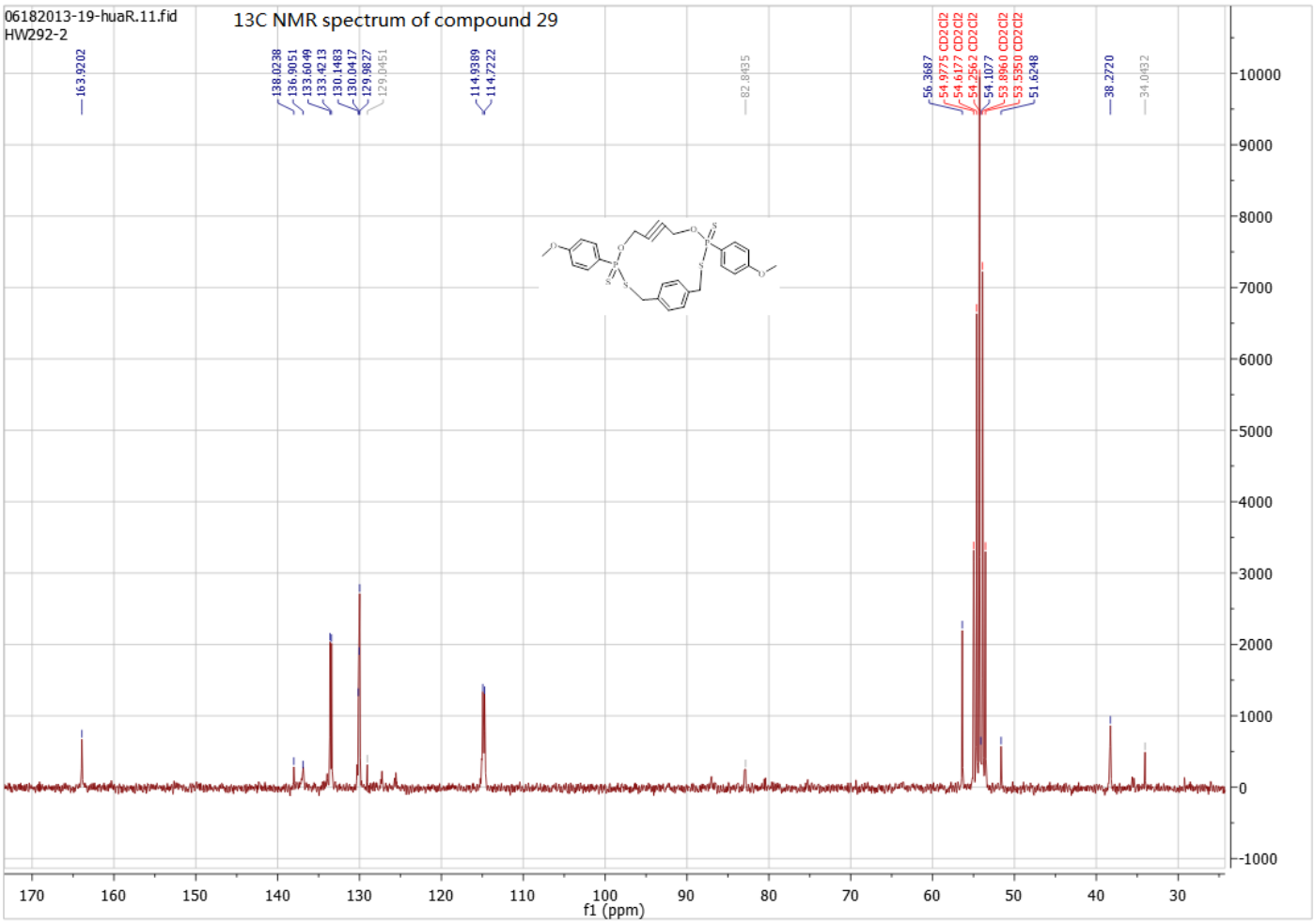

1H NMR spectrum of compound 27
f1 (ppm)

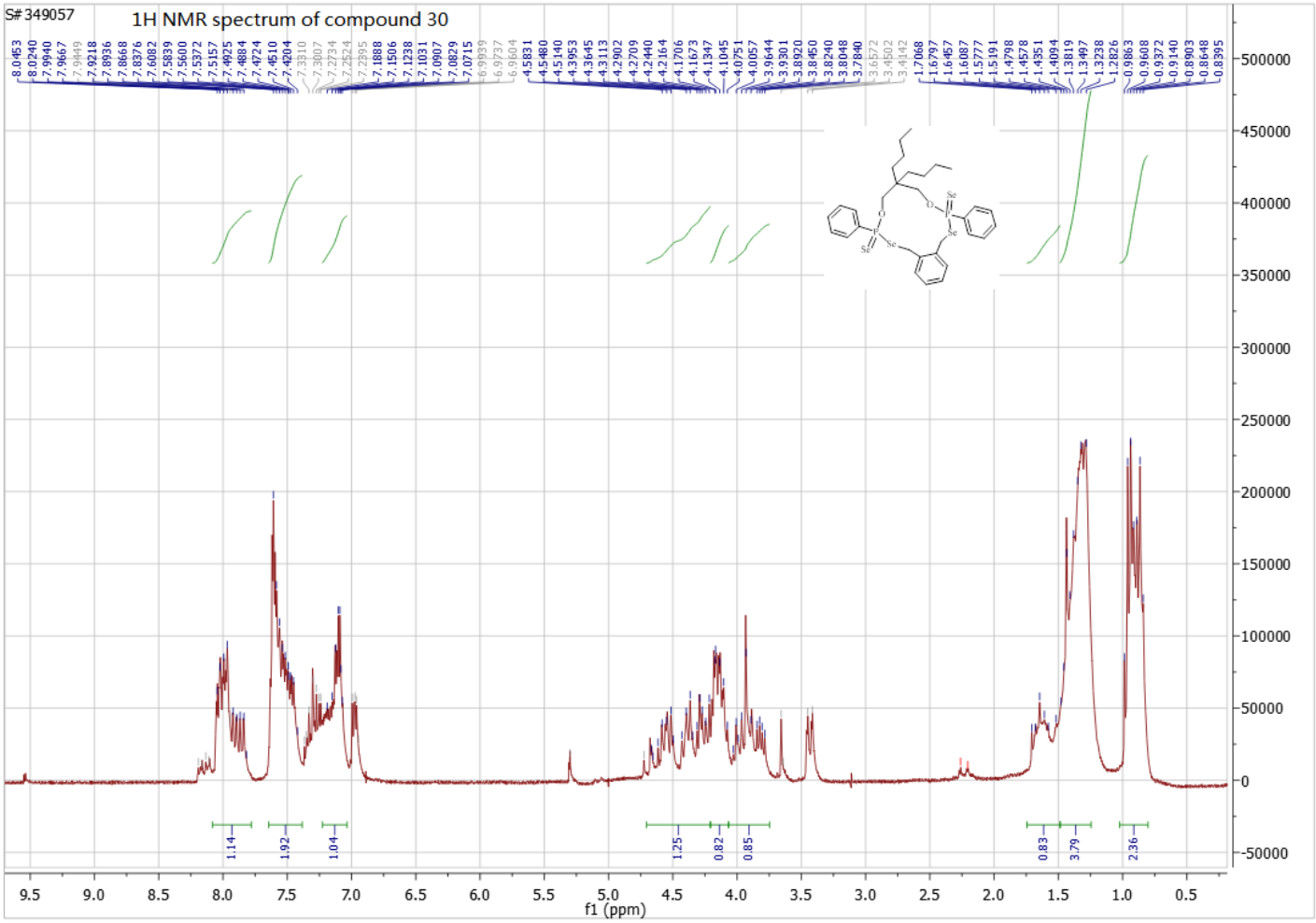

13C NMR spectrum of compound 27
f1 (ppm)

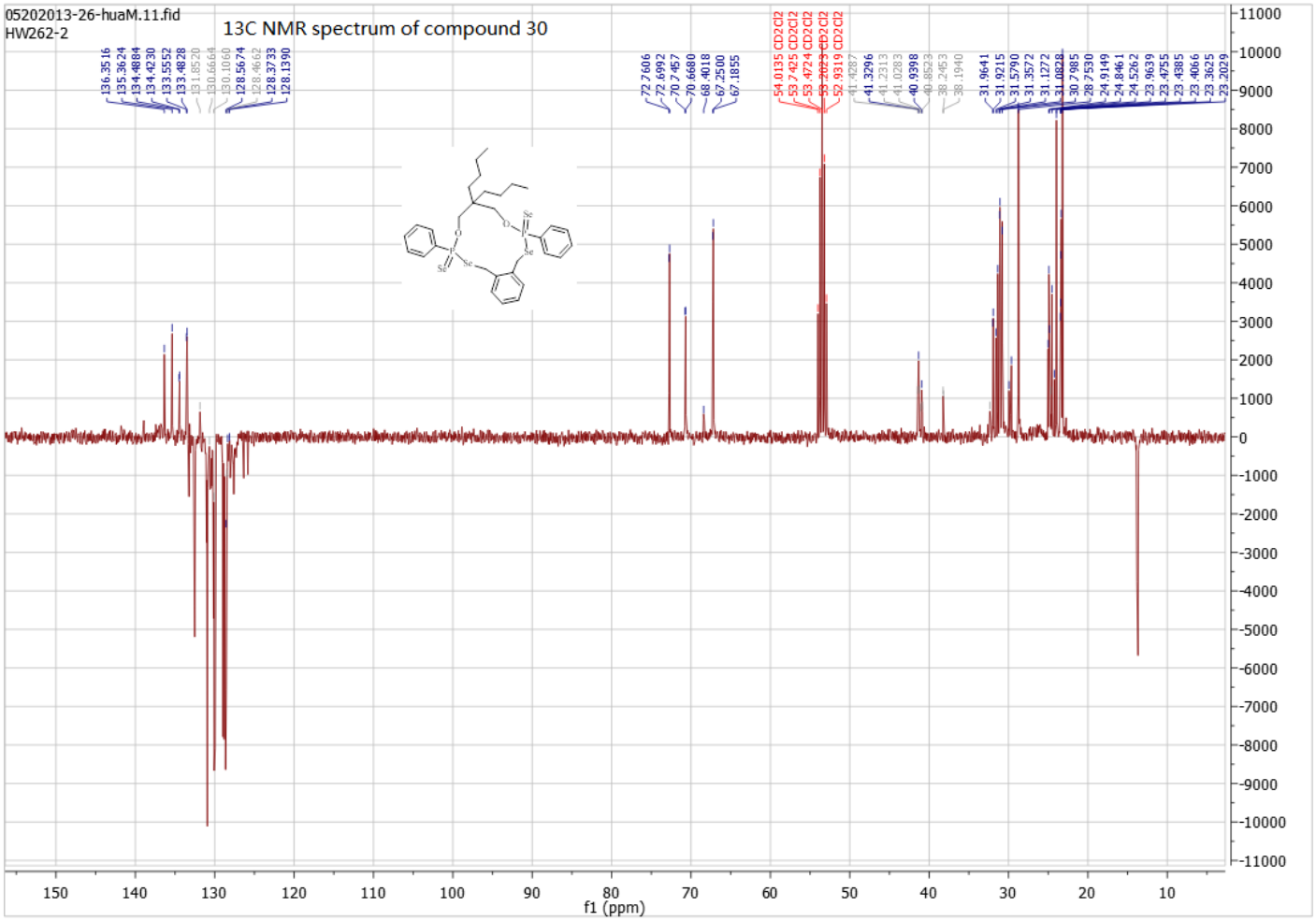

08082013-45-huaM.10.fid
HW298
1H NMR spectrum of compound 28
f1 (ppm)

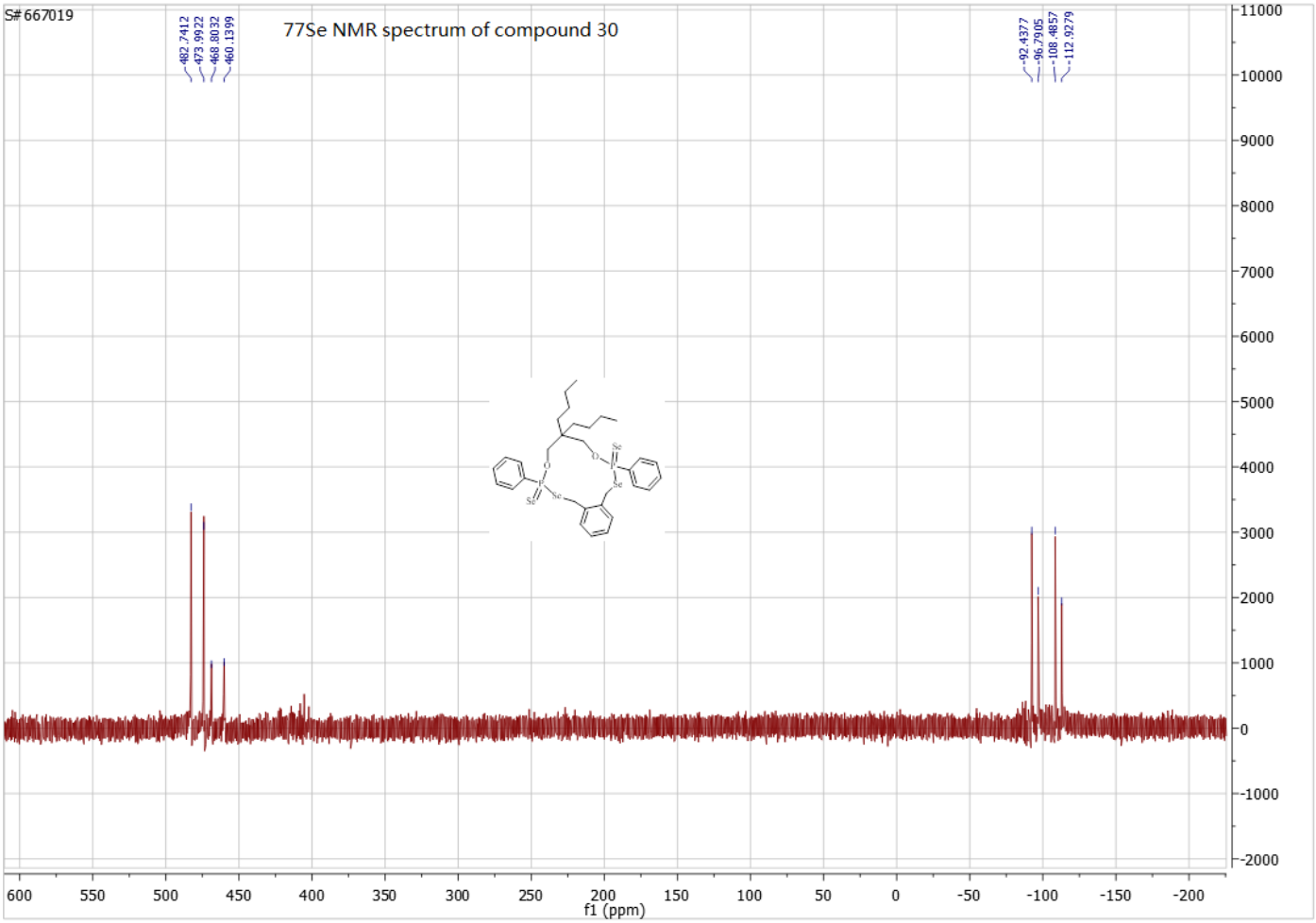

08082013-45-huaM.11.fid
HW298
13C NMR spectrum of compound 28
f1 (ppm)

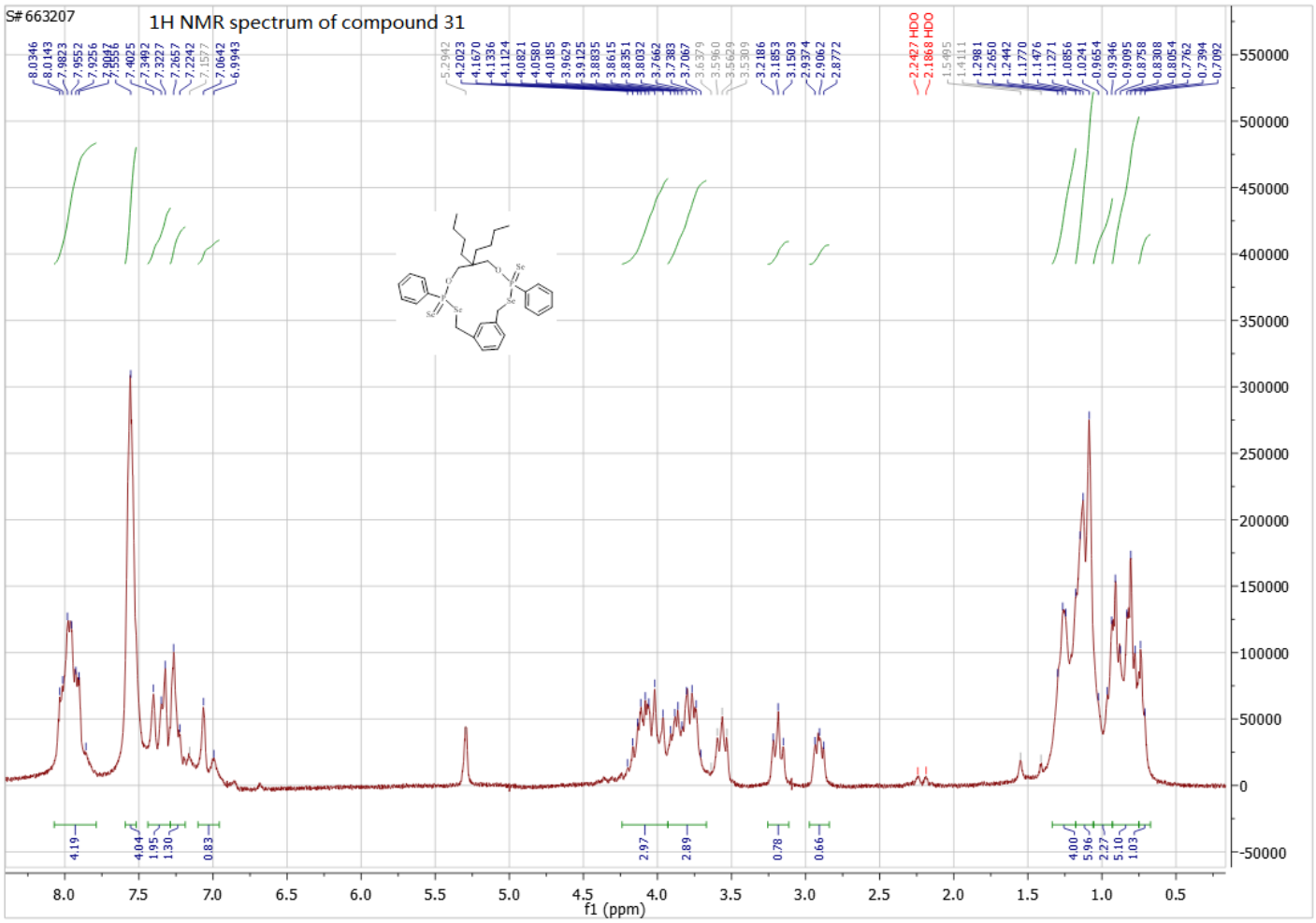

06182013-19-huaR.10.fid
HW292-2
1H NMR spectrum of compound 29
f1 (ppm)

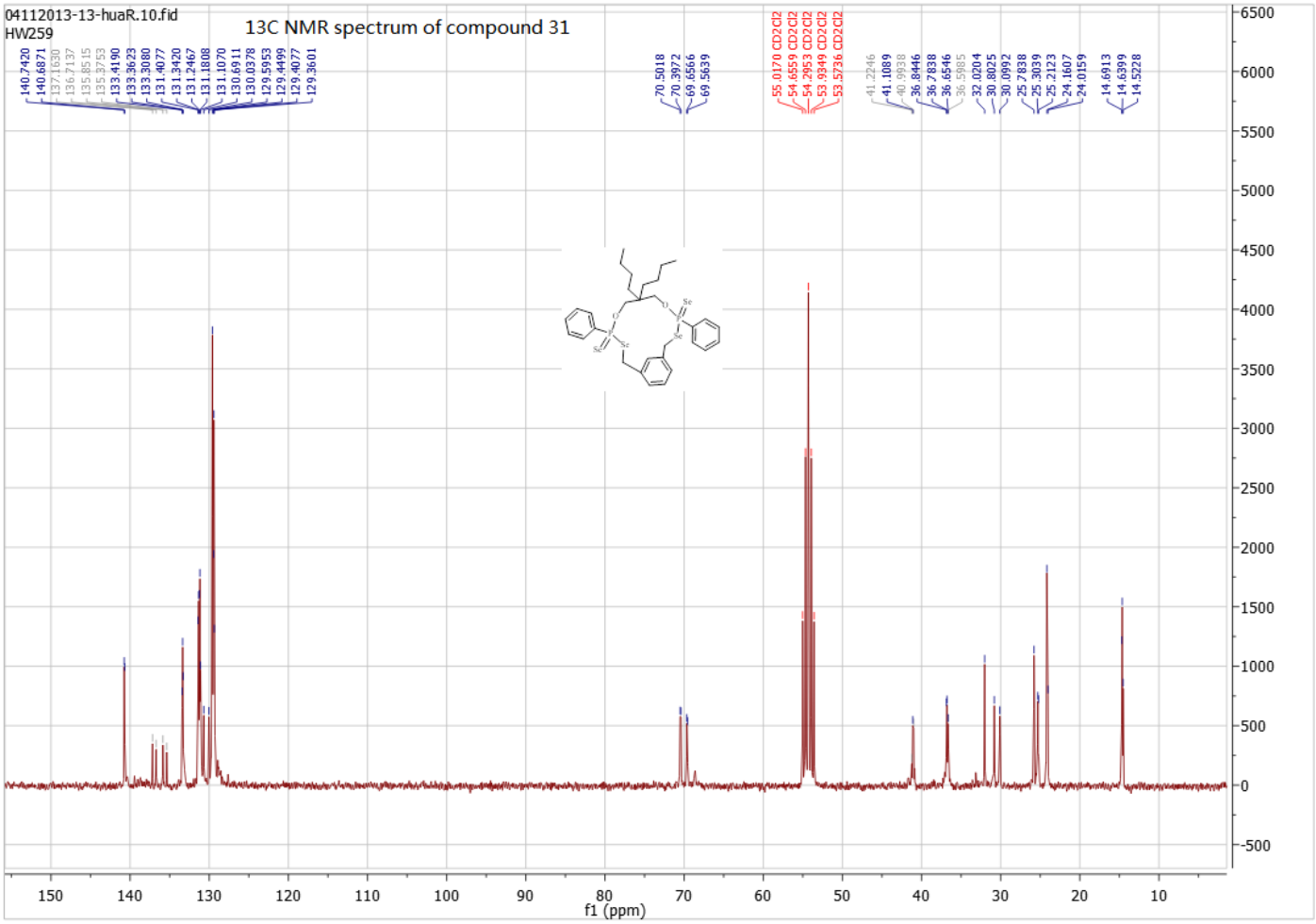

06182013-19-huaR.11.fid
HW292-2
13C NMR spectrum of compound 29
163.9202
138.0238
136.9051
133.6049
133.4213
130.1483
130.0417
129.9827
129.0451
114.9389
114.7222
82.8435
56.3687
54.9775 CD2Cl2
54.6177 CD2Cl2
54.2562 CD2Cl2
54.1077
53.8960 CD2Cl2
53.5350 CD2Cl2
51.6248
38.2720
34.0432
f1 (ppm)

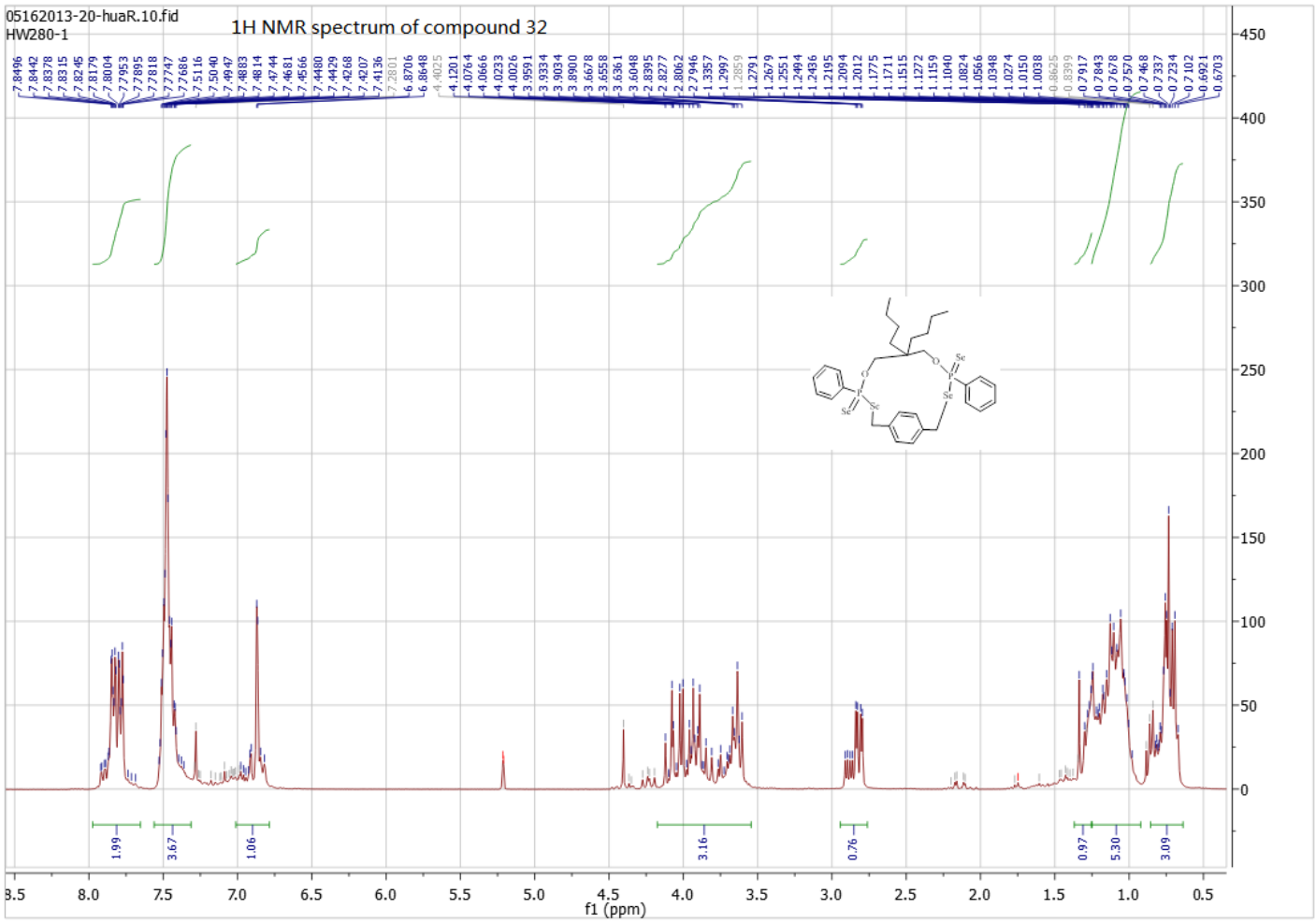

S# 349057
1H NMR spectrum of compound 30
f1 (ppm)

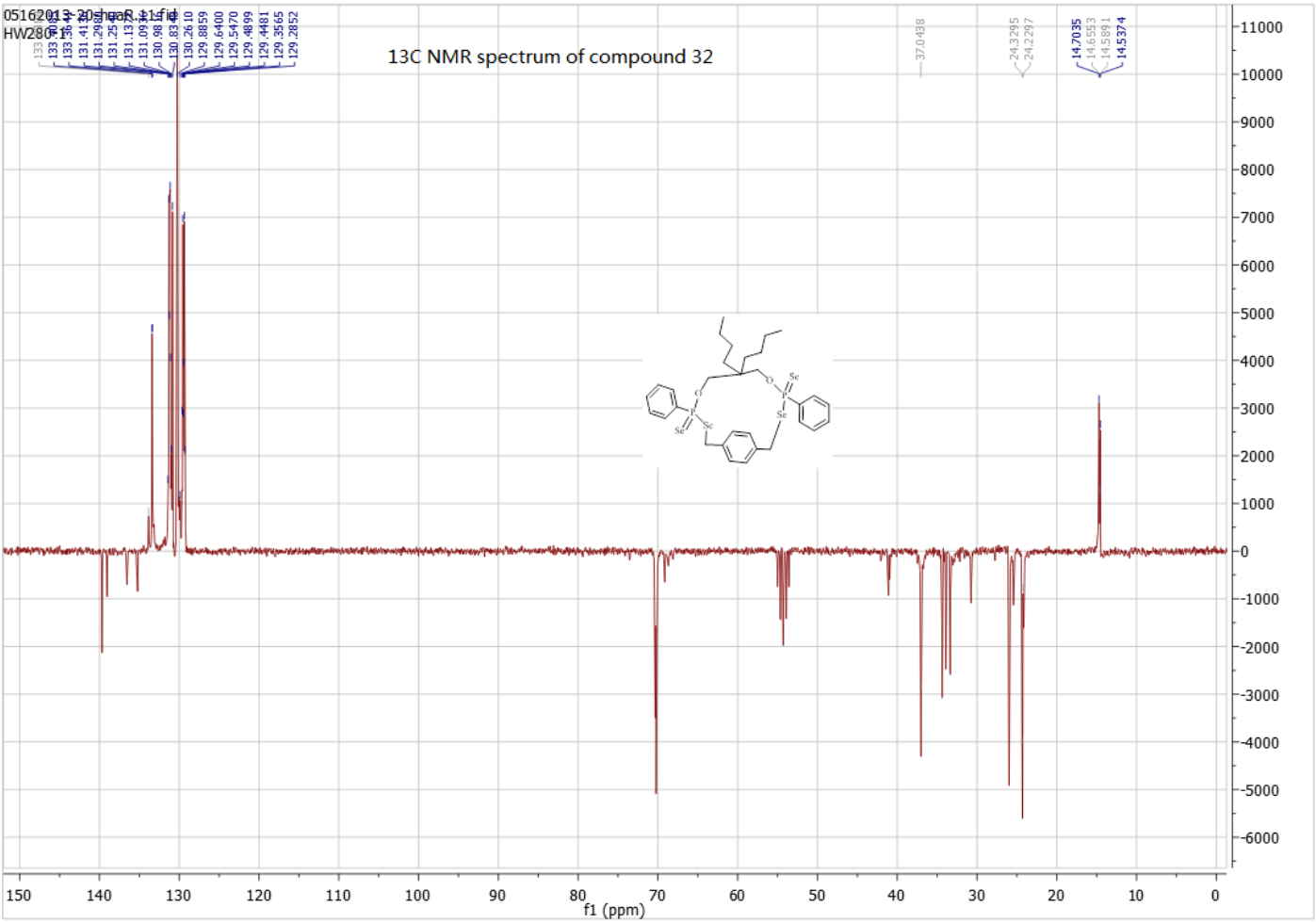

05202013-26-huaM.11.fid
HW262-2
13C NMR spectrum of compound 30
f1 (ppm)

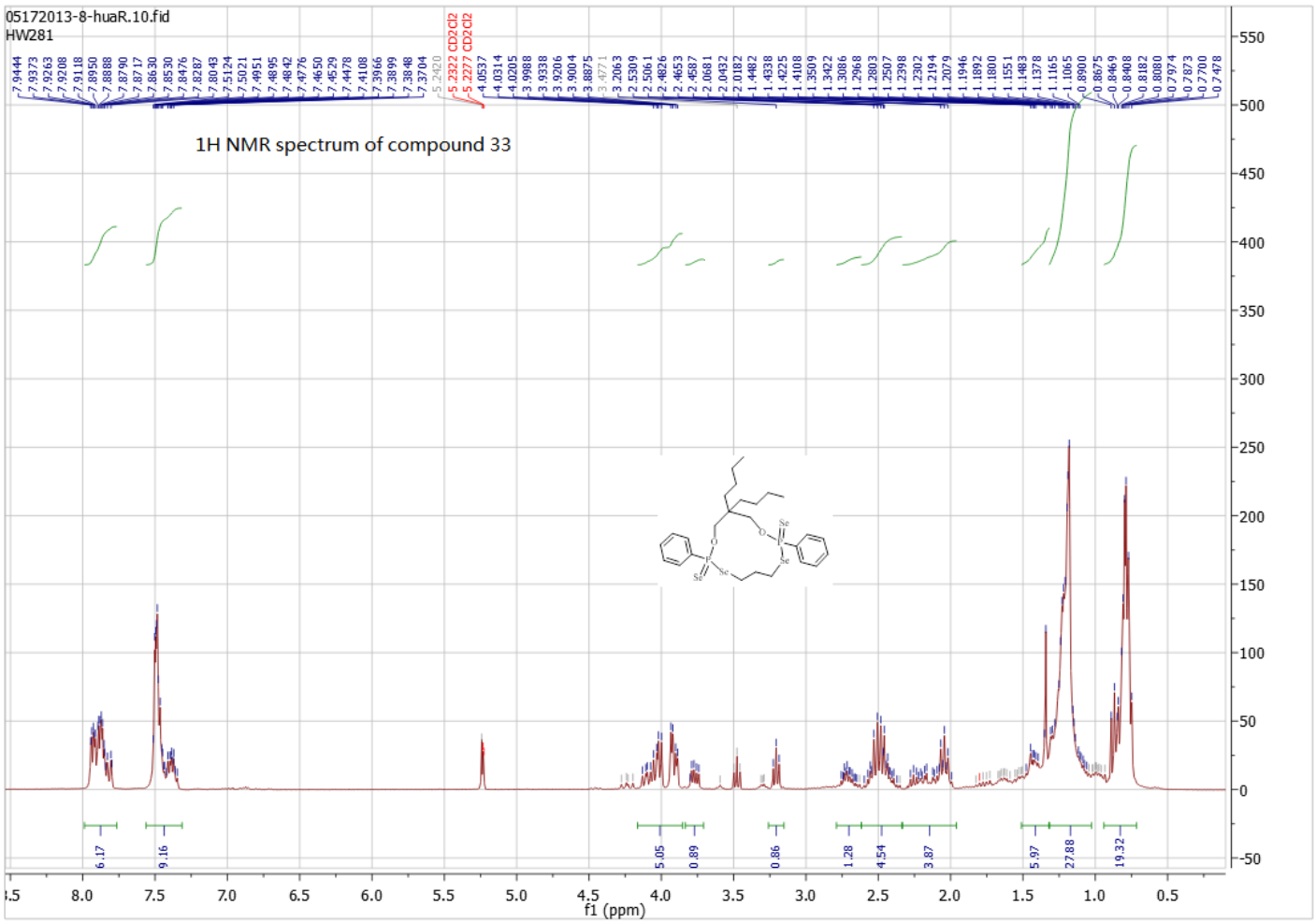

S# 667019
77Se NMR spectrum of compound 30
482.7412
473.9922
468.8032
460.1399
-92.4377
-96.7905
-108.4857
-112.9279
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
600
550
500
450
400
350
300
250
200
150
100
50
0
-50
-100
-150
-200
f1 (ppm)

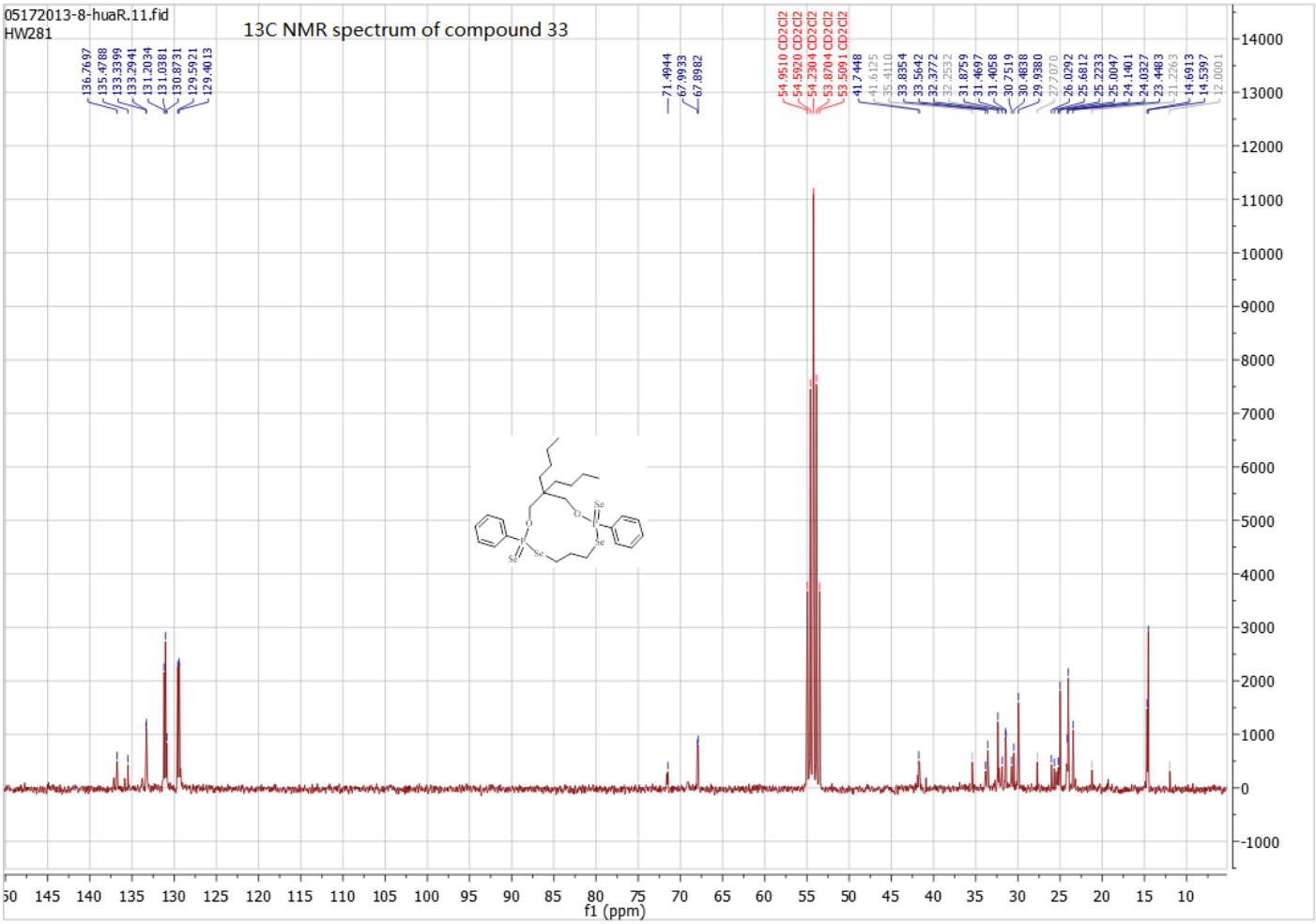

S# 663207
1H NMR spectrum of compound 31
f1 (ppm)

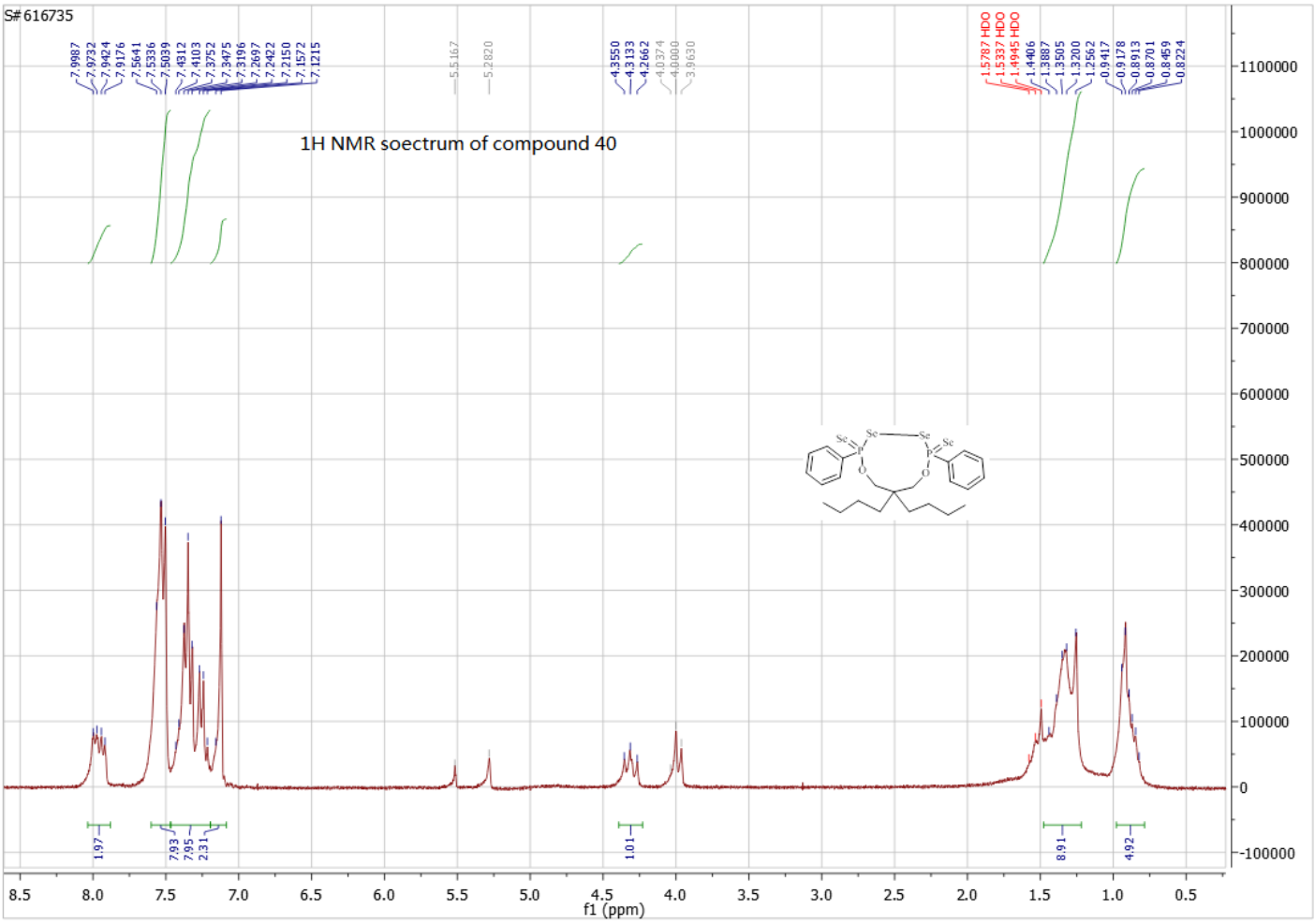

04112013-13-huaR.10.fid
HW259
13C NMR spectrum of compound 31
140.7420
140.6871
137.1630
136.7137
135.8515
135.3753
133.4190
133.3623
133.3080
131.4077
131.3420
131.2467
131.1808
131.1070
130.6911
130.0378
129.5953
129.4499
129.4077
129.3601
70.5018
70.3972
69.6566
69.5639
55.0170 CD2Cl2
54.6559 CD2Cl2
54.2953 CD2Cl2
53.9349 CD2Cl2
53.5736 CD2Cl2
41.2246
41.1089
40.9938
36.8446
36.7838
36.6546
36.5985
32.0204
30.8025
30.0992
25.7838
25.3039
25.2123
24.1607
24.0159
14.6913
14.6399
14.5228
f1 (ppm)

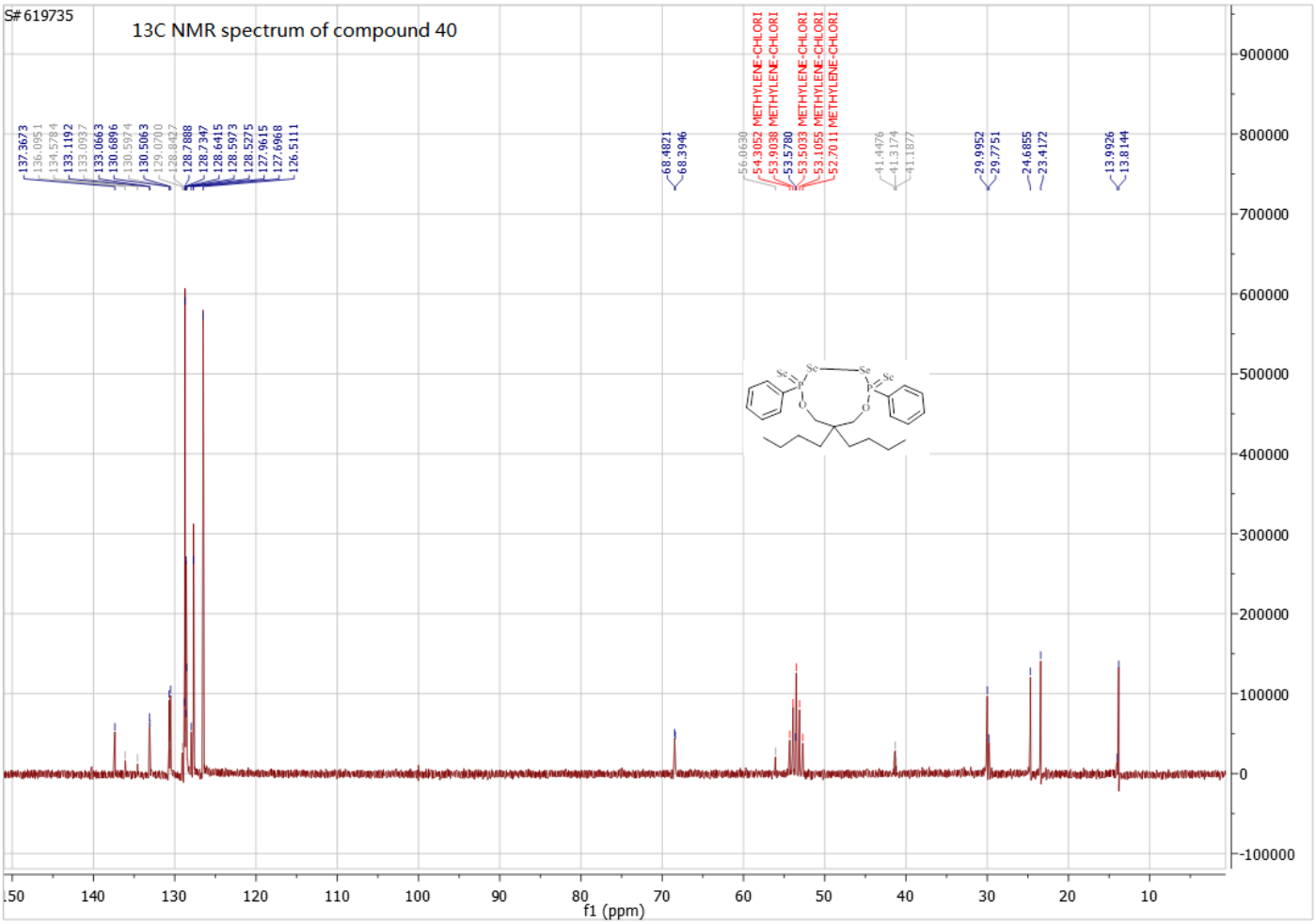

05162013-20-huaR.10.fid
HW280-1
1H NMR spectrum of compound 32
f1 (ppm)

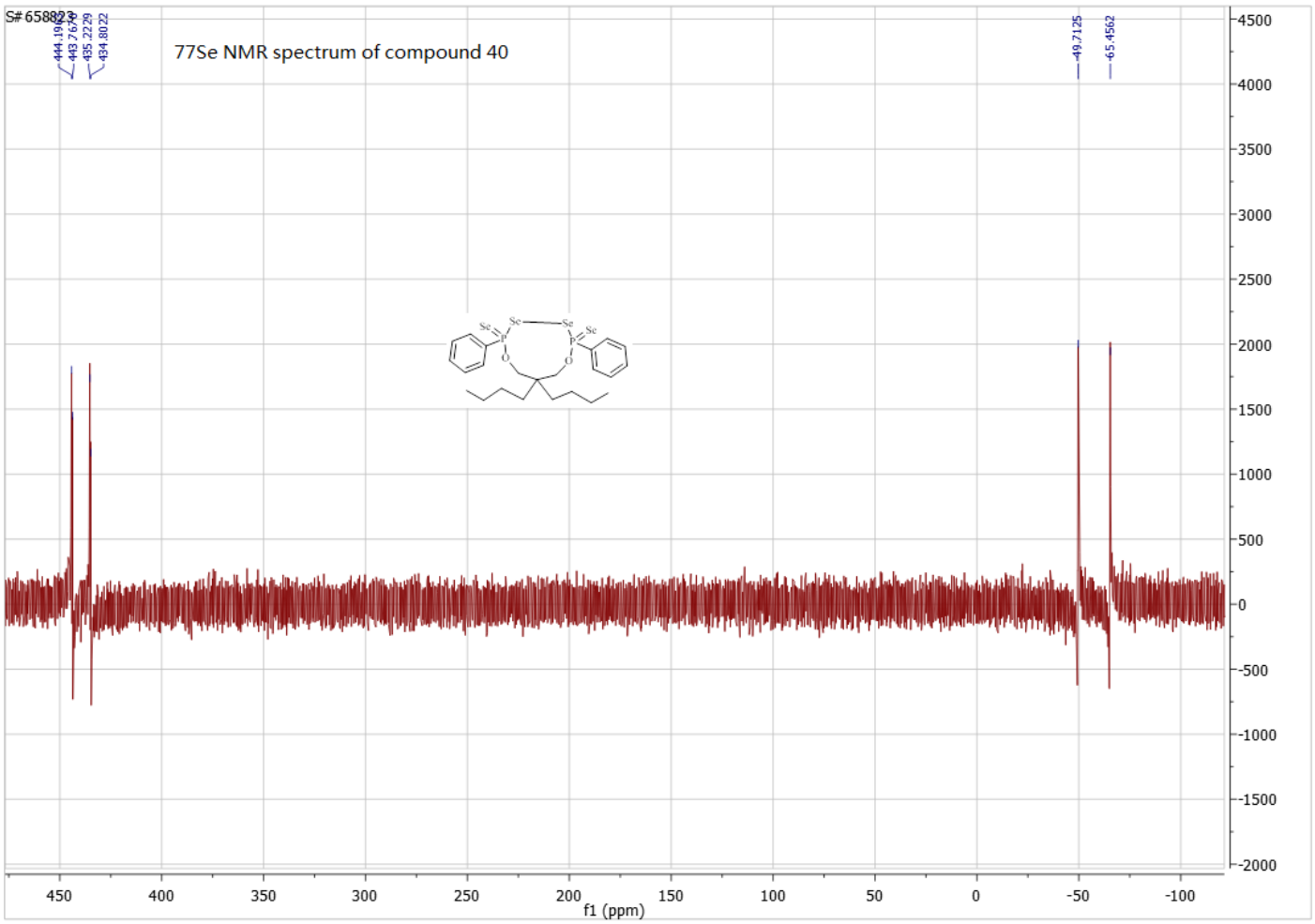

13C NMR spectrum of compound 32
f1 (ppm)


05172013-8-huaR.10.fid
HW281
1H NMR spectrum of compound 33
f1 (ppm)


05172013-8-huaR.11.fid
HW281
13C NMR spectrum of compound 33
136.7697
135.4788
133.3399
133.2941
131.2034
131.0381
130.8731
129.5921
129.4013
71.4944
67.9933
67.8982
54.9510 CD2Cl2
54.5920 CD2Cl2
54.2304 CD2Cl2
53.8704 CD2Cl2
53.5091 CD2Cl2
41.7448
41.6125
35.4110
33.8354
33.5642
32.3772
32.2532
31.8759
31.4697
31.4058
30.7519
30.4838
29.9380
27.2070
26.0292
25.6812
25.2233
25.0047
24.1401
24.0327
23.4483
21.2263
14.6913
14.5397
12.0001
f1 (ppm)


S#616735
1H NMR soectrum of compound 40
f1 (ppm)


S# 619735
13C NMR spectrum of compound 40
137.3673
136.0951
134.5784
133.1192
133.0937
133.0663
130.6896
130.5974
130.5063
129.0700
128.8427
128.7888
128.7347
128.6415
128.5973
128.5275
127.9615
127.6968
126.5111
68.4821
68.3946
56.0630
54.3052 METHYLENE-CHLORI
53.9038 METHYLENE-CHLORI
53.5780
53.5033 METHYLENE-CHLORI
53.1055 METHYLENE-CHLORI
52.7011 METHYLENE-CHLORI
41.4476
41.3174
41.1877
29.9952
29.7751
24.6855
23.4172
13.9926
13.8144
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
-100000
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


S# 658823
77Se NMR spectrum of compound 40
444.1984
443.7674
435.2229
434.8022
49.7125
-65.4562
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
-1000
-1500
-2000
450
400
350
300
250
200
150
100
50
0
-50
-100
f1 (ppm)