Supporting Information

Practical chromatography-free synthesis of 2-iodo-*N*,*N*-diisopropyl-ferrocenecarboxamide and further transformations

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Pictures of large scale reactions mixtures and TLC

Synthesis of ferrocenecarboxylic acid (3)

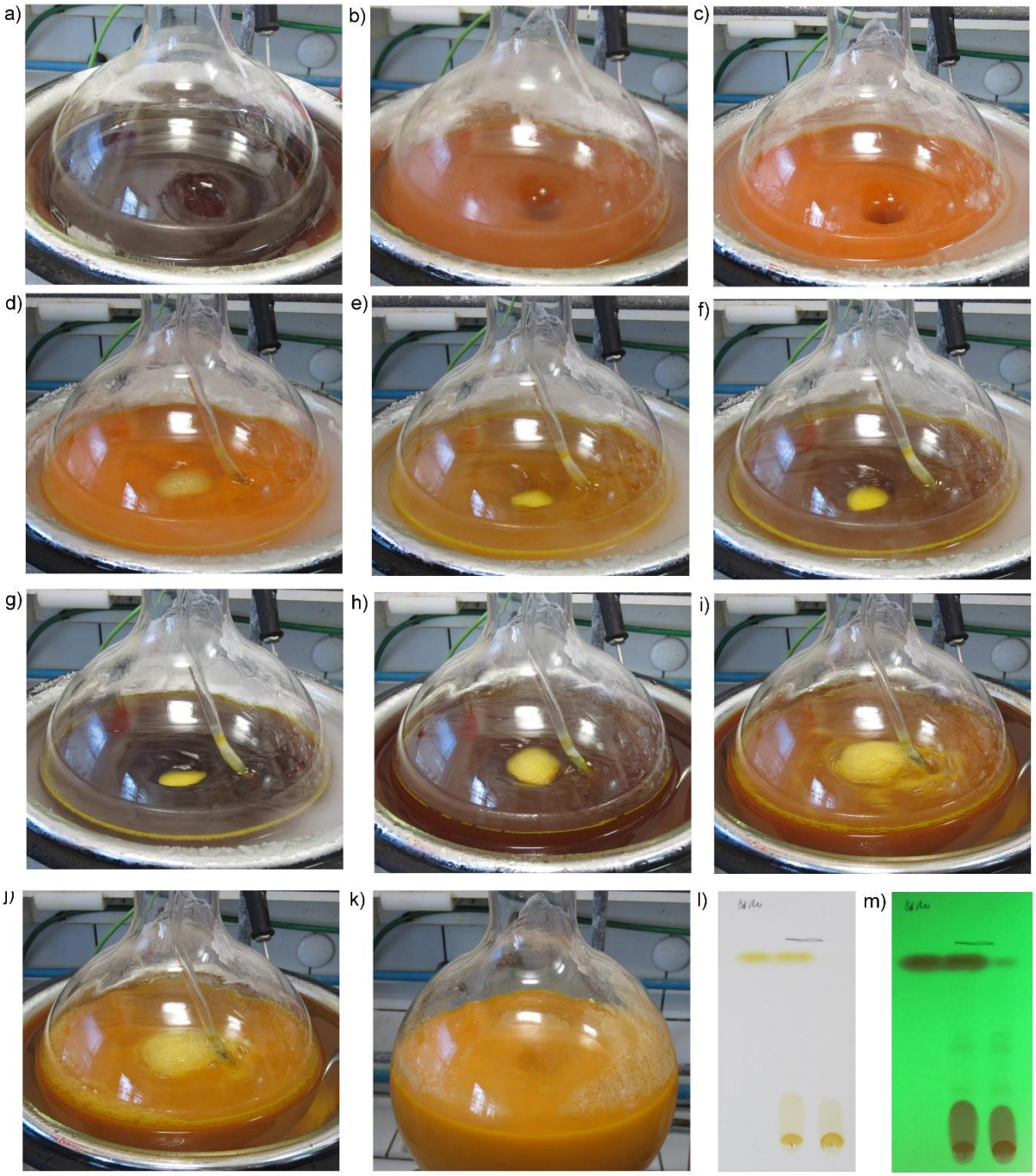


Figure 1. a) Reaction mixture before addition of *t*BuLi; b) Reaction mixture during addition of *t*BuLi; c) Reaction mixture 1 h after addition of *t*BuLi; d) Reaction mixture at the beginning of CO_2 bubbling; e-g) Reaction mixture during CO_2 bubbling; h-i) Reaction mixture warming to rt; j) Reaction mixture warming to rt; k) Reaction mixture when reaction is complete; l) TLC under visible light, PET/EtOAc (80:20); m) TLC under UV light, PET/EtOAc (80:20).

Synthesis of *N*,*N*-diisopropylferrocenecarboxamide (1)

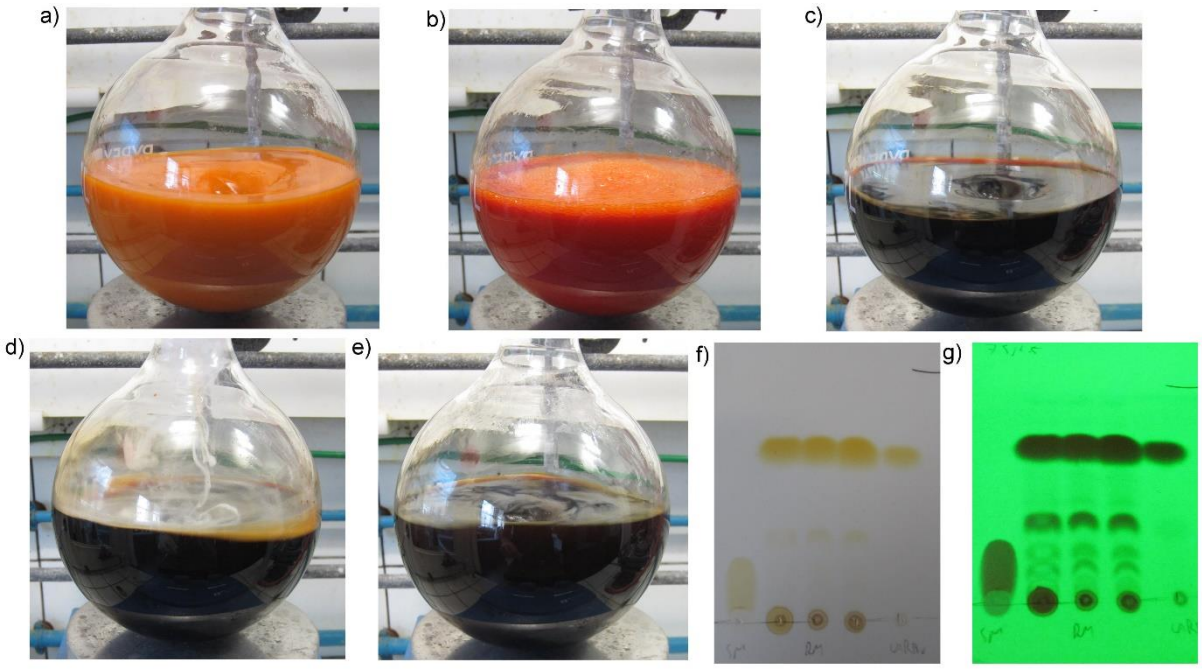


Figure 2. a) Reaction mixture before addition of oxalyl chloride; b) Reaction mixture during and after addition of oxalyl chloride; c) Reaction mixture 30 min after addition of oxalyl chloride; d) Reaction mixture during addition of diisopropylamine; e) Reaction mixture 30 min after addition of diisopropylamine; f) TLC under visible light, PET/EtOAc (75:25); g) TLC under UV light, PET/EtOAc (75:25).

Synthesis of (±)-2-iodo-*N*,*N*-diisopropylferrocenecarboxamide ((±)-2) by using *n*BuLi-TMEDA

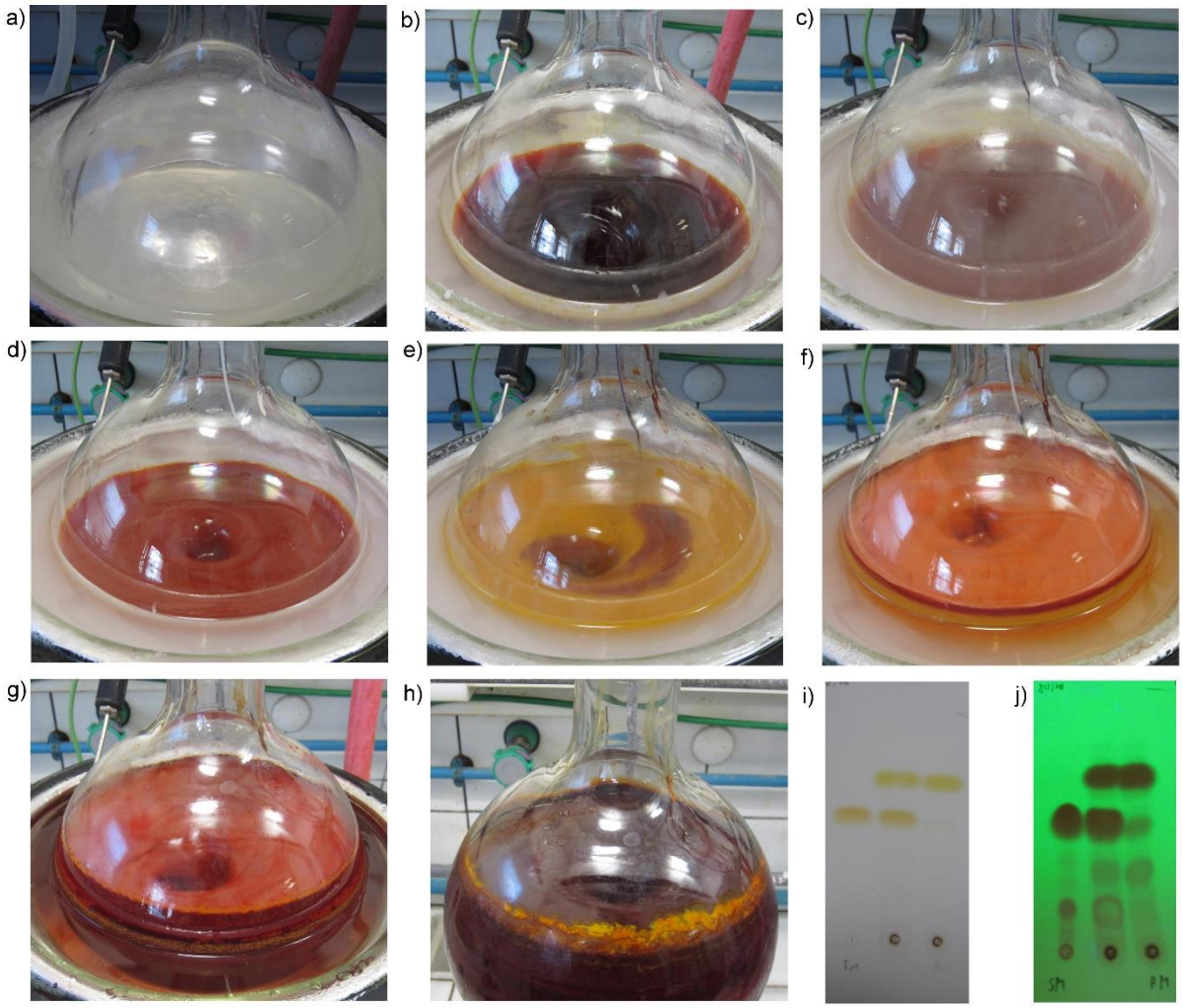


Figure 3. a) The *n*BuLi/TMEDA chelate solution; b) Reaction mixture 1 h after addition of **1**; c-e) Reaction mixture during addition of iodine; f) Reaction mixture after addition of iodine; g-h) Reaction mixture upon warming to rt; i) TLC under visible light, PET/EtOAc (80:20); j) TLC under UV light, PET/EtOAc (80:20).

Synthesis of (±)-2-iodo-*N*,*N*-diisopropylferrocenecarboxamide ((±)-2) by using LiTMP-$ZnCl_2$

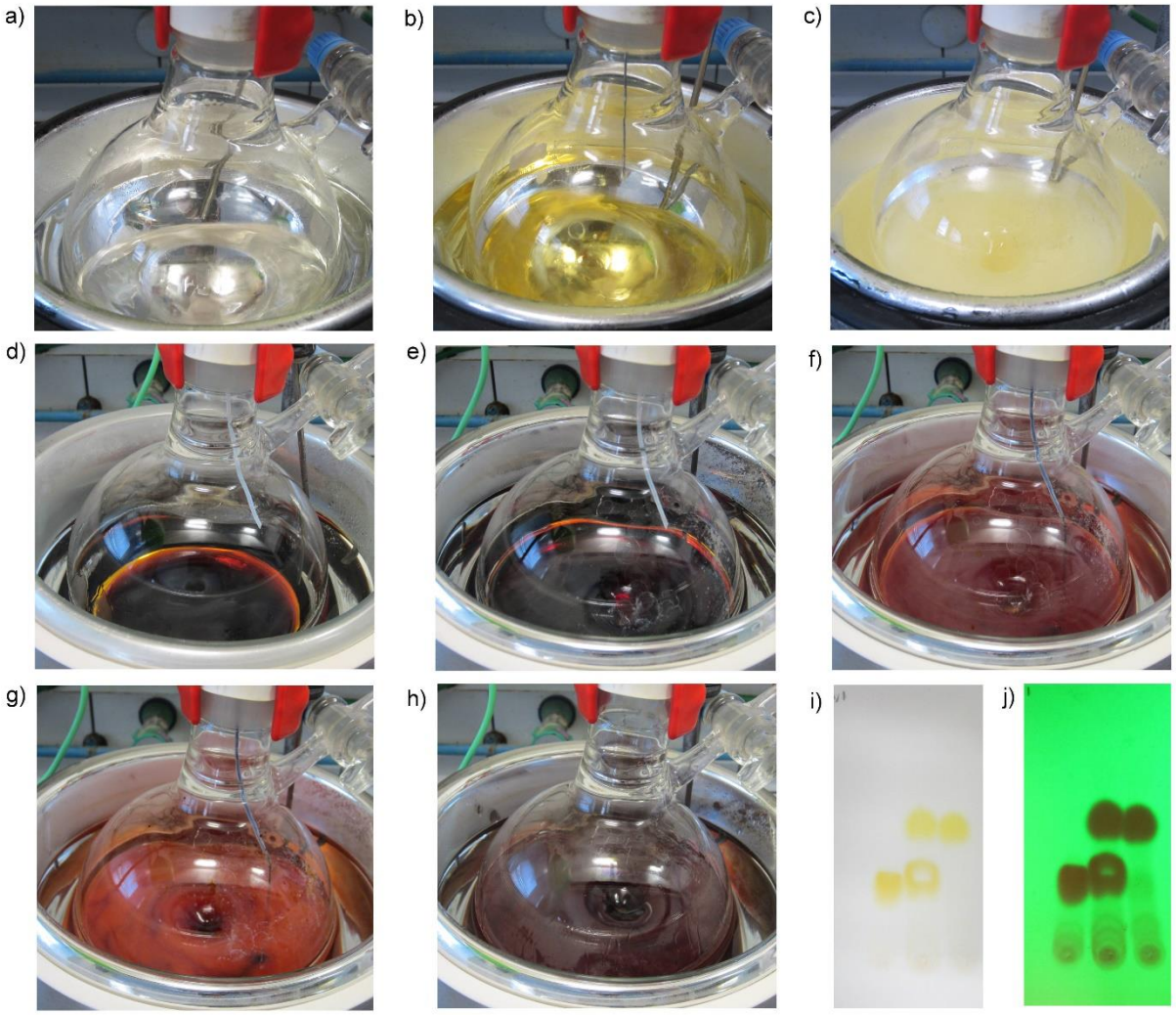


Figure 4. a) The TMPH solution in THF; b) Reaction mixture upon addition of *n*BuLi; c) Reaction mixture 10 min after addition of *n*BuLi; d) The compound **1** and $ZnCl_2$ solution in THF; e) Reaction mixture 2 h after addition of LiTMP solution; f-g) Reaction mixture upon addition of iodine in solution in THF; h) Reaction mixture after addition of iodine; i) TLC under visible light, PET/EtOAc (90:10); j) TLC under UV light, PET/EtOAc (90:10).

Synthesis of (S_p)-2-iodo-*N*,*N*-diisopropylferrocenecarboxamide ((S_p)-2)

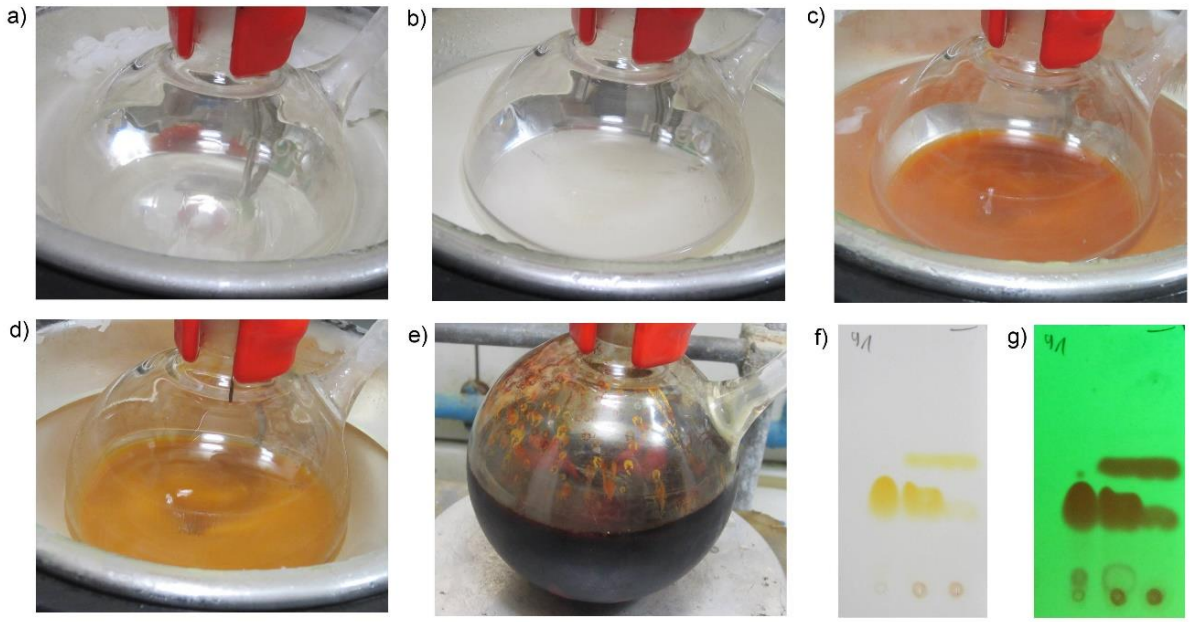


Figure 5. a) The (+)-sparteine surrogate solution; b) The *n*BuLi/(+)-sparteine surrogate chelate solution; c) Reaction mixture 1 h after addition of **1**; d) Reaction mixture during addition of iodine; e) Reaction mixture once at rt; f) TLC under visible light, PET/EtOAc (90:10); g) TLC under UV light, PET/EtOAc (90:10).

Crystallography

For **(S_p)-2**, **(±)-4**, **(±)-5**, **(±)-6**, **(±)-7**, **(±)-9**, **(±)-10**, **(±)-11**, **(±)-13**, **(±)**-14 and **(±)-17**, the X-ray diffraction data were collected using D8 VENTURE Bruker AXS diffractometer at the temperature given in the crystal data. The samples were studied with monochromatized Mo-Kα radiation (λ = 0.71073 Å). The structure was solved by dual-space algorithm using the *SHELXT* program,[1] and then refined with full-matrix least-square methods based on F^2 (*SHELXL*).[2] All non-hydrogen atoms were refined with anisotropic atomic displacement parameters. H atoms were finally included in their calculated positions and treated as riding on their parent atom with constrained thermal parameters. The molecular diagrams were generated by MERCURY (version 3.9).

Crystal data for (S_p)-2. $C_{17}H_{22}FeINO$, M = 439.10, T = 150 K; orthorhombic $P\,2_1\,2_1\,2_1$ (I.T.#19), a = 11.317(2), b = 13.283(3), c = 23.793(6) Å, V = 3576.4(15) Å^3. Z = 8, d = 1.631 g.cm^{-3}, μ = 2.568 mm^{-1}. A final refinement on F^2 with 7918 unique intensities and 334 parameters converged at ω$R(F^2)$ = 0.1162 ($R(F)$ = 0.0537) for 5744 observed reflections with $I > 2\sigma(I)$. CCDC 1898624.

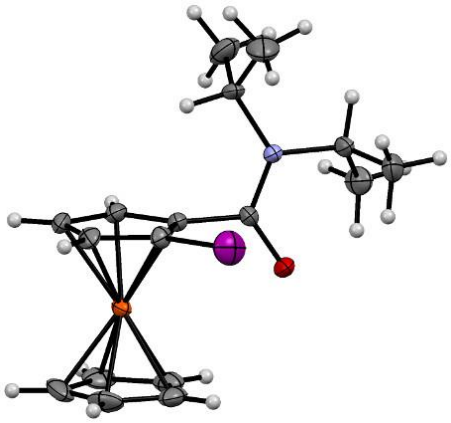


Figure 6. Molecular structure of compound **(S_p)-2** (thermal ellipsoids shown at the 30% probability level).

Crystal data for (±)-4. $C_{23}H_{27}FeNO$, M = 389.30, T = 150 K; monoclinic $P\,2_1/n$ (I.T.#14), a = 14.9375(19), b = 18.213(2), c = 15.7162(19) Å, β = 111.799(4) °, V = 3969.9(9) Å^3. Z = 8, d = 1.303 g.cm^{-3}, μ = 0.770 mm^{-1}. A final refinement on F^2 with 9016 unique intensities and 477 parameters converged at ω$R(F^2)$ = 0.0689 ($R(F)$ = 0.0323) for 7434 observed reflections with $I > 2\sigma(I)$. CCDC 1909912.

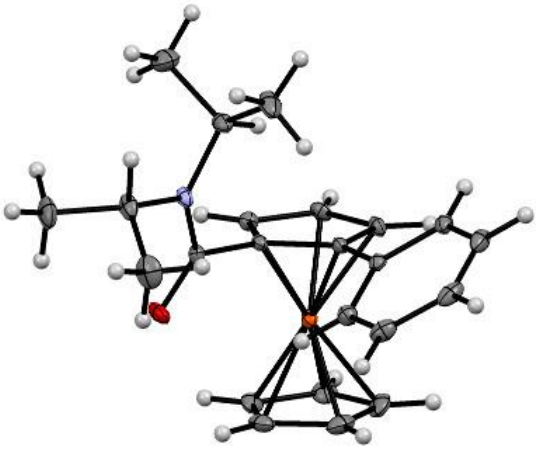


Figure 7. Molecular structure of compound **(±)-4** (thermal ellipsoids shown at the 30% probability level).

Crystal data for (±)-5. $C_{24}H_{29}FeNO_2$, M = 419.33, T = 150 K; monoclinic $C\,c$ (I.T.#9), a = 13.0954(12), b = 21.838(2), c = 7.2746(8) Å, β = 90.262(4) °, V = 2080.4(4) Å^3. Z = 4, d = 1.339 g.cm^{-3}, μ = 0.744 mm^{-1}. A final refinement on F^2 with 4445 unique intensities and 241 parameters converged at ωR(F^2) = 0.0787 (R(F) = 0.0355) for 4206 observed reflections with $I > 2\sigma(I)$. CCDC 1909910.

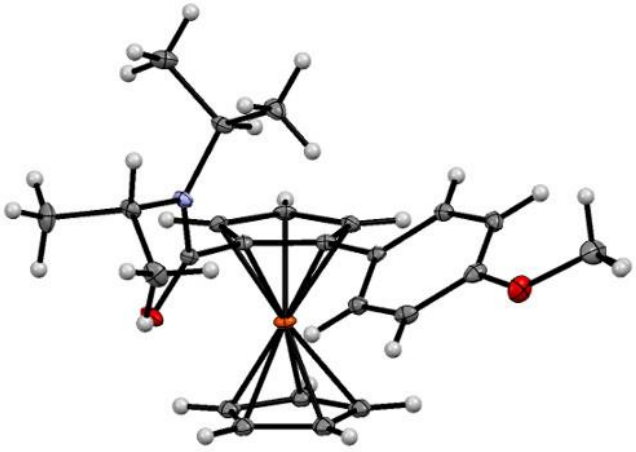


Figure 8. Molecular structure of compound **(±)-5** (thermal ellipsoids shown at the 30% probability level).

Crystal data for (±)-6. $C_{25}H_{31}FeNO_3$, M = 449.36, T = 150 K; monoclinic $P\,2_1/c$ (I.T.#14), a = 10.5791(11), b = 10.6613(11), c = 19.8541(18) Å, β = 91.090(4) °, V = 2238.9(4) Å^3. Z = 4, d = 1.333 g.cm^{-3}, μ = 0.699 mm^{-1}. A final refinement on F^2 with 5095 unique intensities and 277 parameters converged at ωR(F^2) = 0.0802 (R(F) = 0.0328) for 4440 observed reflections with $I > 2\sigma(I)$. CCDC 1909911.

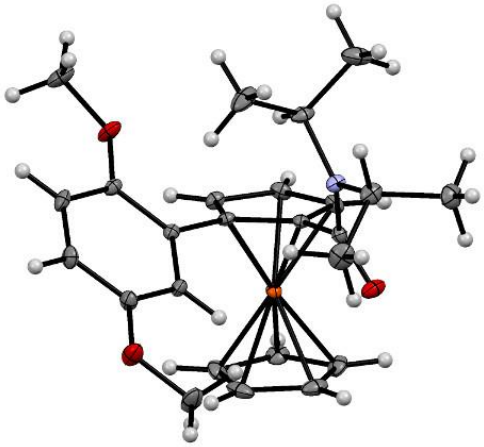


Figure 9. Molecular structure of compound **(±)-6** (thermal ellipsoids shown at the 30% probability level).

Crystal data for (±)-7. $C_{27}H_{29}FeNO$, M = 439.36, T = 150 K; monoclinic C 2/c (I.T.#15), a = 32.567(4), b = 7.4303(8), c = 22.463(3) Å, β = 125.311(4) °, V = 4435.6(9) Å^3. Z = 8, d = 1.316 g.cm^{-3}, μ = 0.698 mm^{-1}. A final refinement on F^2 with 5072 unique intensities and 275 parameters converged at ωR(F^2) = 0.1041 (R(F) = 0.0396) for 4391 observed reflections with I > 2σ(I). CCDC 1909914.

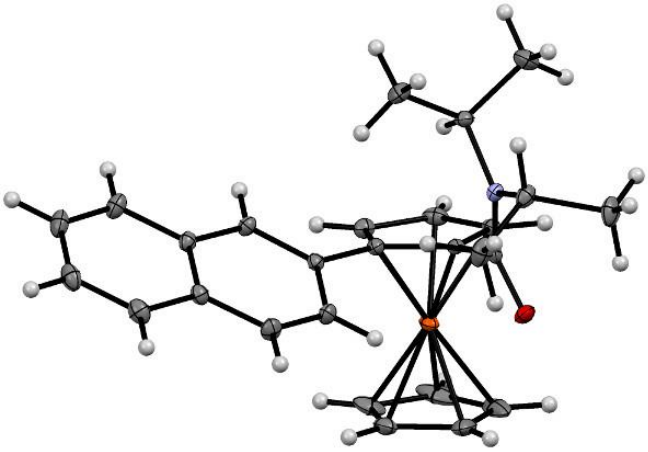


Figure 10. Molecular structure of compound **(±)-7** (thermal ellipsoids shown at the 30% probability level).

Crystal data for (±)-9. $C_{23}H_{26}ClFeNO$, M = 423.75, T = 150 K; monoclinic P 2$_1$/c (I.T.#14), a = 14.084(2), b = 10.914(2), c = 13.538(2) Å, β = 103.597(6) °, V = 2022.7(6) Å^3. Z = 4, d = 1.392 g.cm^{-3}, μ = 0.890 mm^{-1}. A final refinement on F^2 with 4556 unique intensities and 248 parameters converged at ωR(F^2) = 0.0888 (R(F) = 0.0373) for 3764 observed reflections with I > 2σ(I). CCDC 1909913.

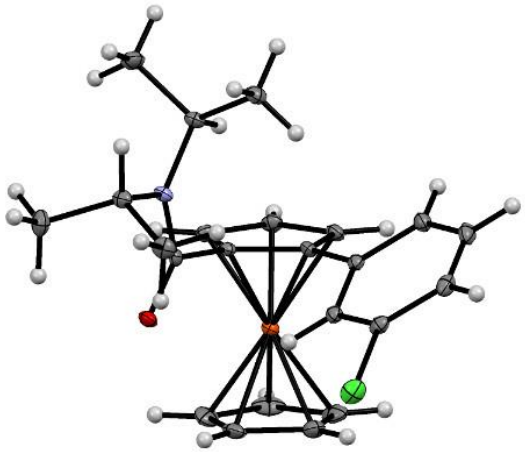


Figure 11. Molecular structure of compound **(±)-9** (thermal ellipsoids shown at the 30% probability level).

Crystal data for (±)-10. $C_{24}H_{26}F_3FeNO$, M = 457.31, T = 150 K; monoclinic $P\,2_1/n$ (I.T.#14), a = 11.8596(11), b = 10.9447(9), c = 16.5793(15) Å, β = 89.930(4) °, V = 2152.0(3) Å^3. Z = 4, d = 1.411 g.cm^{-3}, μ = 0.741 mm^{-1}. A final refinement on F^2 with 4888 unique intensities and 273 parameters converged at ω$R(F^2)$ = 0.1613 ($R(F)$ = 0.0667) for 4028 observed reflections with $I > 2\sigma(I)$. CCDC 1909915.

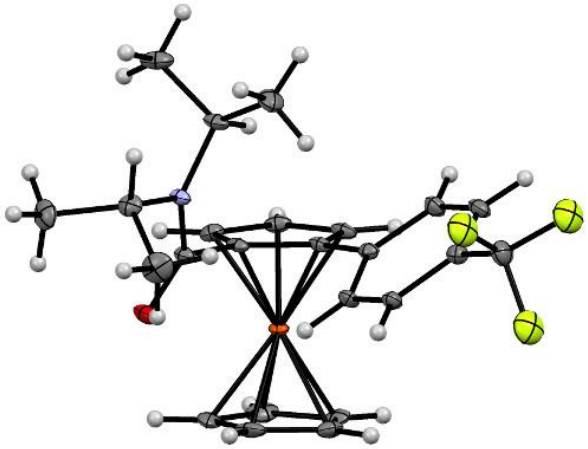


Figure 12. Molecular structure of compound **(±)-10** (thermal ellipsoids shown at the 30% probability level).

Crystal data for (±)-11. $C_{24}H_{26}FFeNO_3$, M = 451.31, T = 150 K; triclinic P -1 (I.T.#2), a = 13.1191(17), b = 13.2958(15), c = 14.9573(16) Å, α = 71.416(4), β = 65.243(4), γ = 89.744(4) °, V = 2220.3(5) Å^3. Z = 4, d = 1.350 g.cm^{-3}, μ = 0.712 mm^{-1}. A final refinement on F^2 with 10158 unique intensities and 494 parameters converged at ω$R_F{}^2$ = 0.1025 (R_F = 0.0415) for 8397 observed reflections with $I > 2\sigma(I)$. CCDC 1909916.

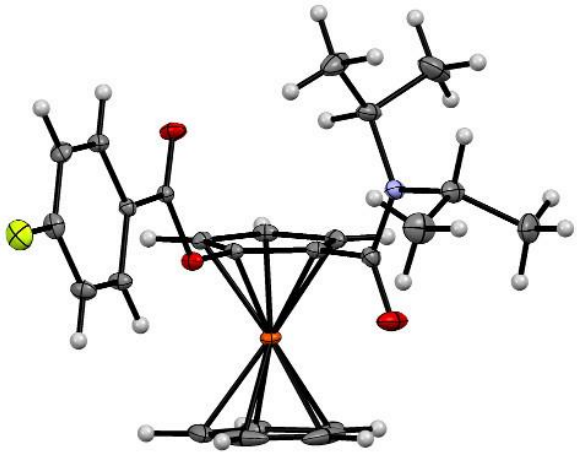


Figure 13. Molecular structure of compound **(±)-11** (thermal ellipsoids shown at the 30% probability level).

Crystal data for (±)-13. $C_{26}H_{31}FeNO_3$, M = 461.37, T = 150 K; monoclinic $P\,2_1/c$ (I.T.#14), a = 13.6489(15), b = 7.5313(6), c = 22.135(2) Å, β = 91.206(4) °, V = 2274.9(4) Å^3. Z = 4, d = 1.347 g.cm^{-3}, μ = 0.690 mm^{-1}. A final refinement on F^2 with 5174 unique intensities and 253 parameters converged at ωR_F^2 = 0.1510 (R_F = 0.0636) for 3967 observed reflections with $I > 2\sigma(I)$. CCDC 1909919.

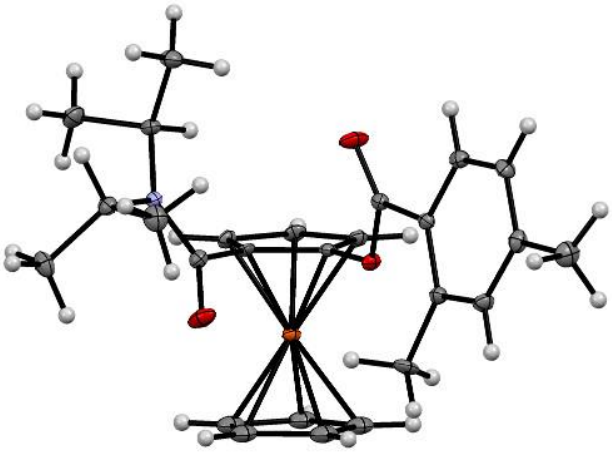


Figure 14. Molecular structure of compound **(±)-13** (thermal ellipsoids shown at the 30% probability level).

Crystal data for (±)-14. $C_{26}H_{29}FeNO_5$, M = 491.35, T = 150 K; triclinic P -1 (I.T.#2), a = 10.4904(12), b = 11.0366(12), c = 11.2048(11) Å, α = 93.771(4), β = 108.488(4), γ = 100.808(4) °, V = 1197.5(2) Å^3. Z = 2, d = 1.363 g.cm^{-3}, μ = 0.666 mm^{-1}. A final refinement on F^2 with 5501 unique intensities and 303 parameters converged at ωR_F^2 = 0.0838 (R_F = 0.0342) for 4987 observed reflections with $I > 2\sigma(I)$. CCDC 1909918.

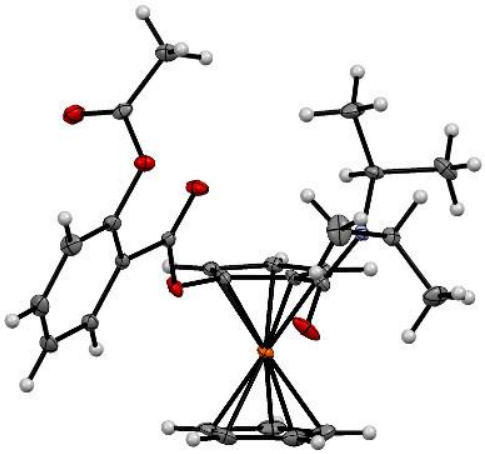


Figure 15. Molecular structure of compound **(±)-14** (thermal ellipsoids shown at the 30% probability level).

Crystal data for (±)-16. $C_{28}H_{31}Fe_2NO_3$, M = 541.24, triclinic P -1 (I.T.#2), a = 7.3553(6), b = 10.6982(11), c = 15.9658(15) Å, α = 91.068(4), β = 93.064(3), γ = 103.447(3) °, V = 1219.5(2) Å^3. Z = 2, d = 1.474 g.cm^{-3}, μ = 1.220 mm^{-1}. A final refinement on F^2 with 5588 unique intensities and 311 parameters converged at ωR_F^2 = 0.0753 (R_F = 0.0274) for 4758 observed reflections with $I > 2\sigma(I)$. CCDC 1909917.

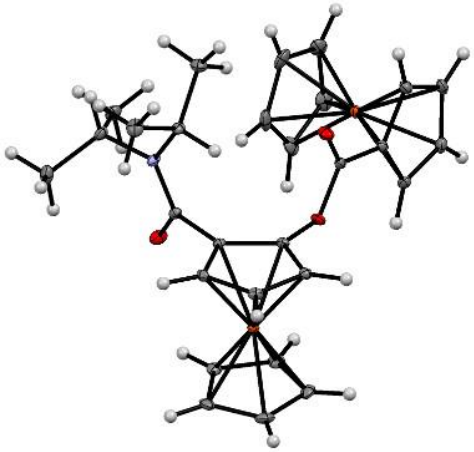
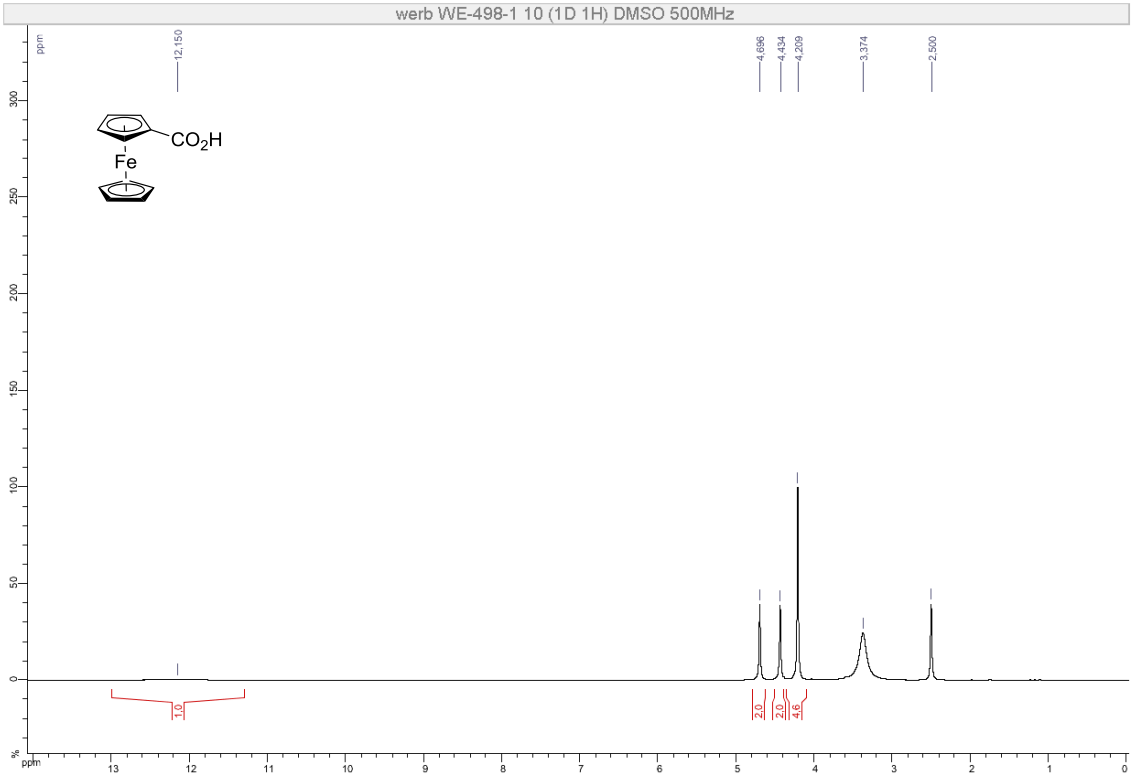
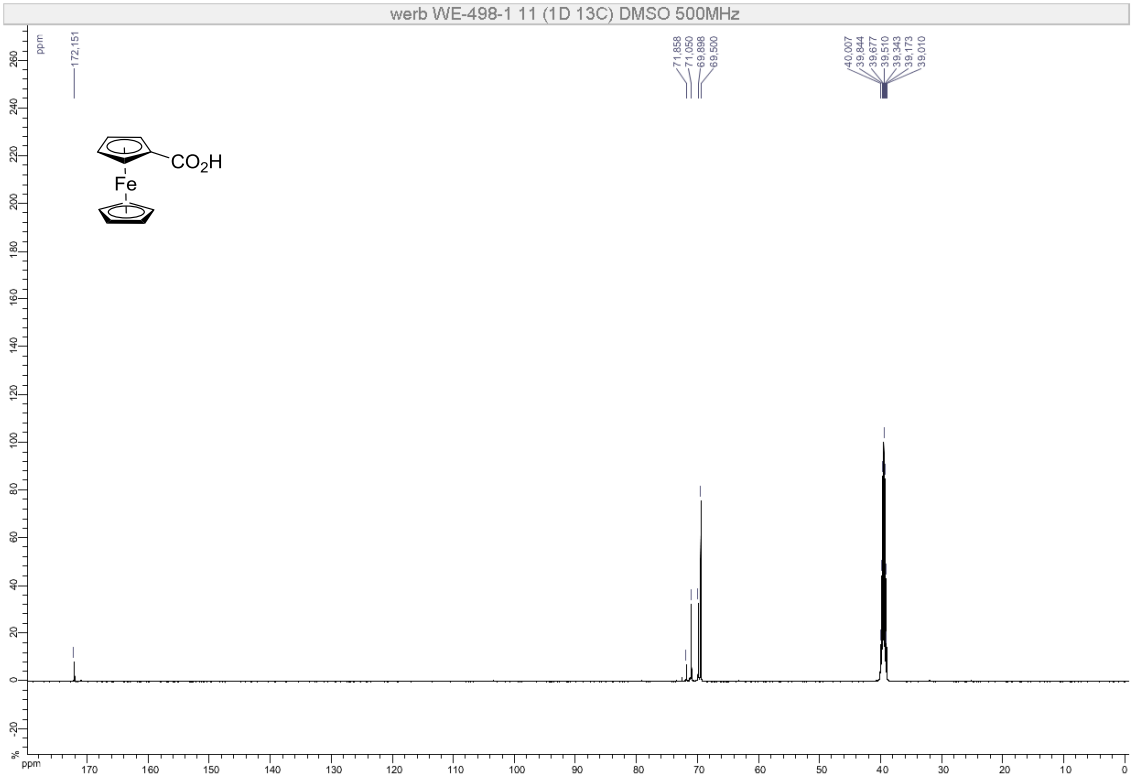


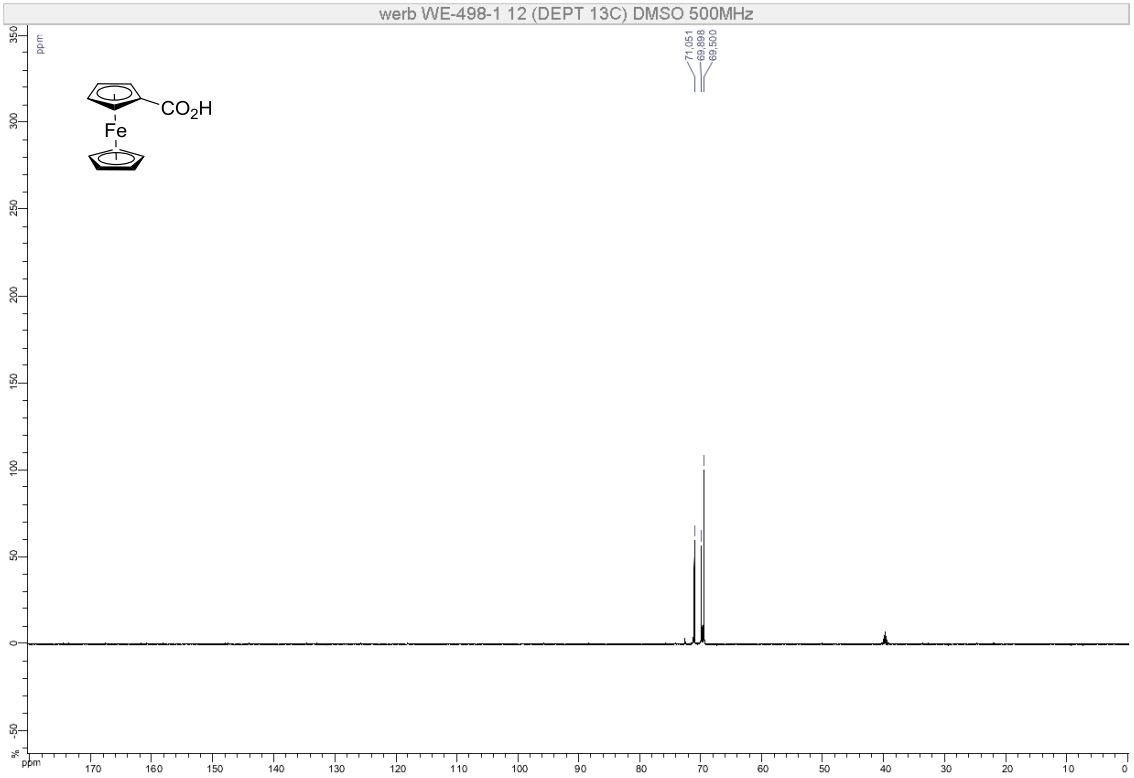
Figure 16. Molecular structure of compound **(±)-16** (thermal ellipsoids shown at the 30% probability level).

NMR Spectra

Compound 3



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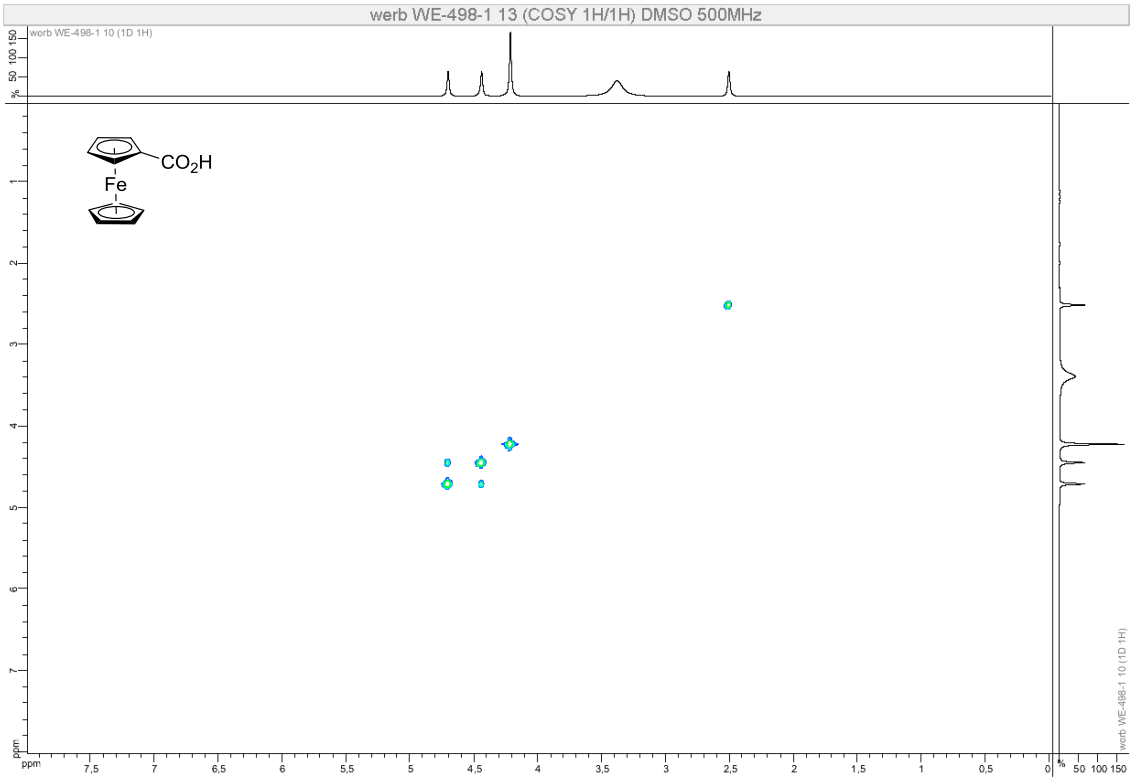
69,898
68,500
CO2H
Fe
ppm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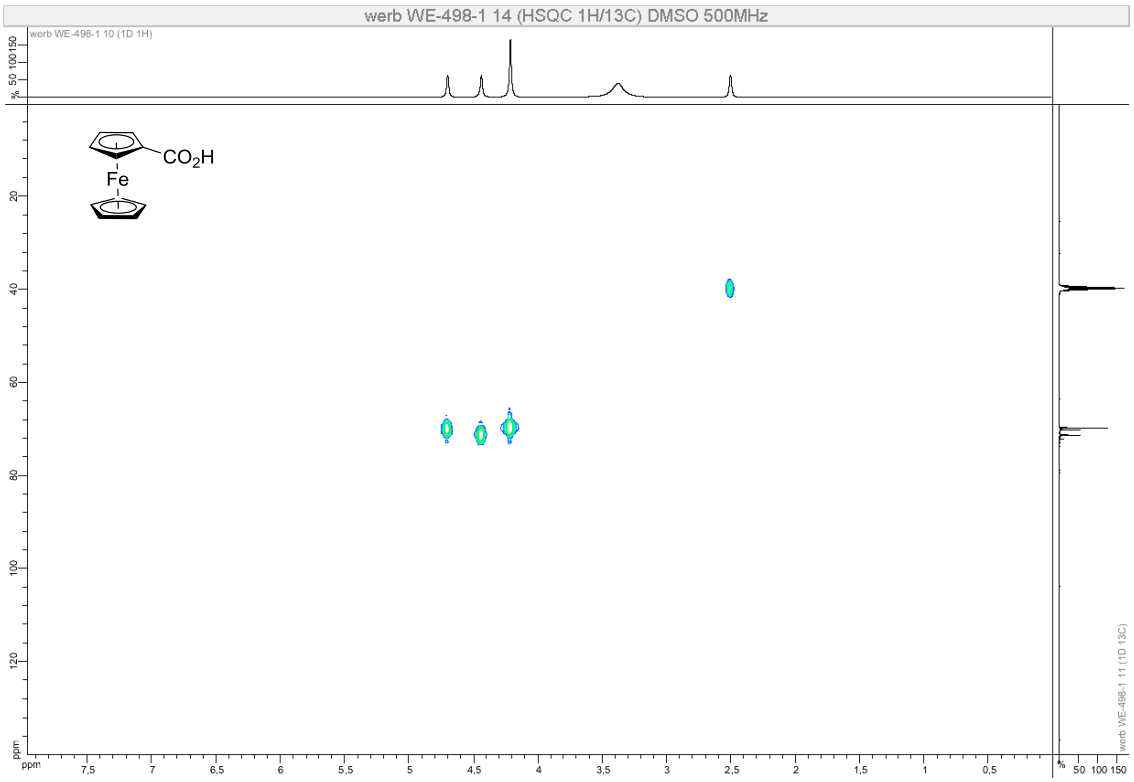
werb WE-498-1 10 (1D 1H)
CO2H
Fe
ppm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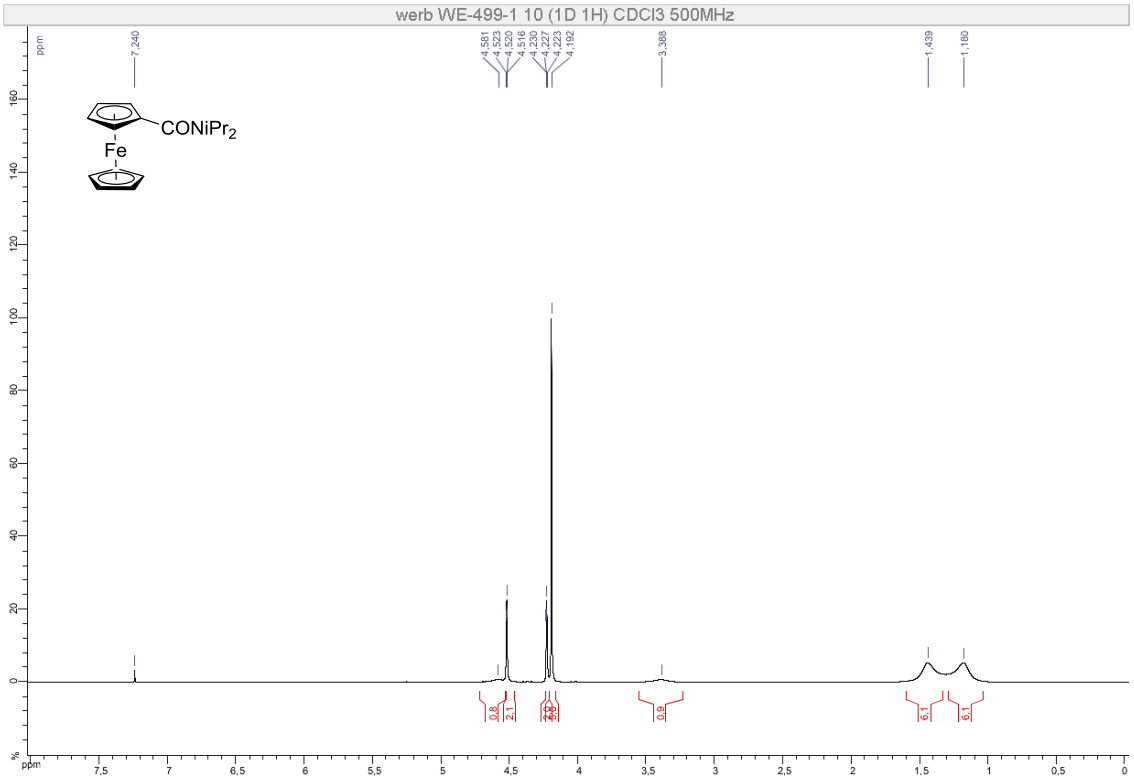
werb WE-498-1 10 (1D 1H)
CO2H
Fe
ppm
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5
werb WE-498-1 11 (1D 13C)

Compound 1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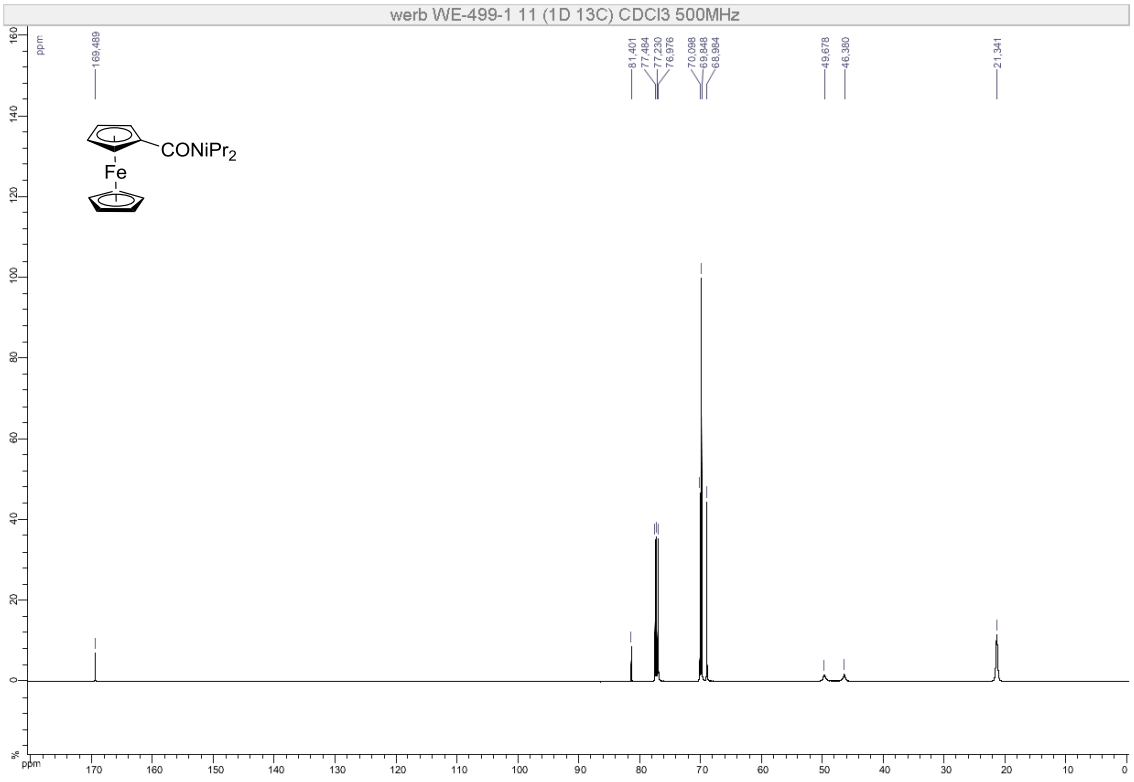
CONiPr2
Fe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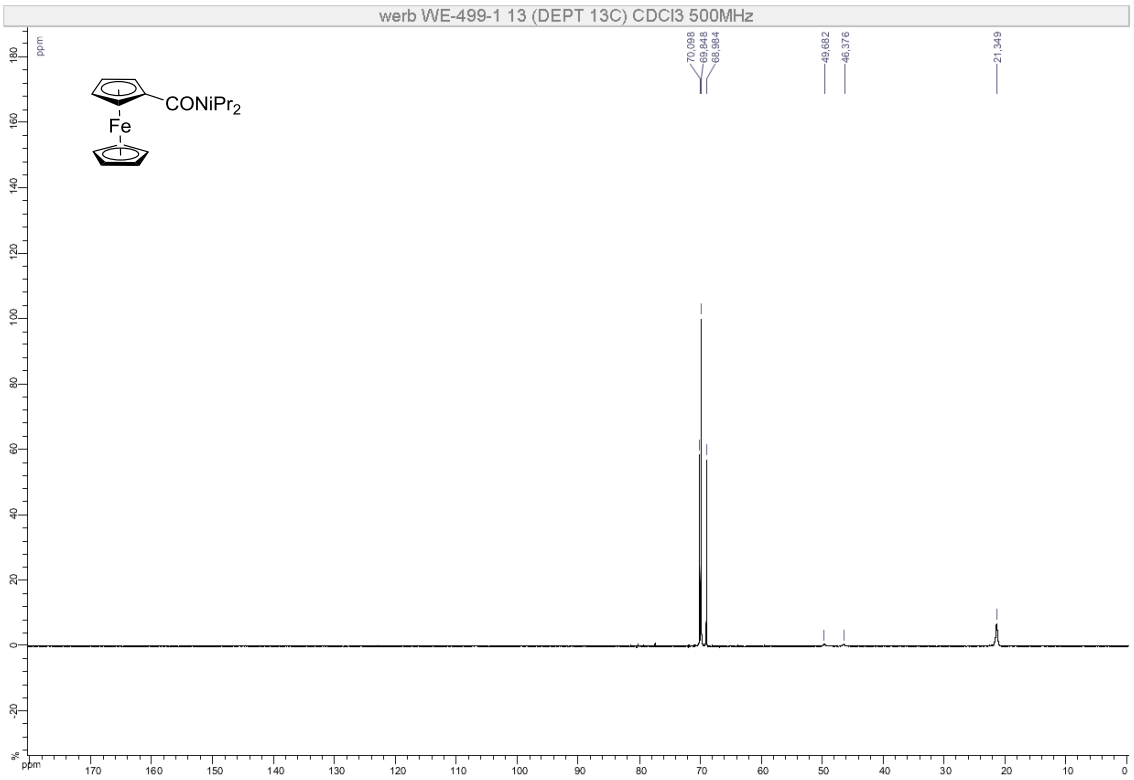
CONiPr2
Fe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CONiPr2
Fe
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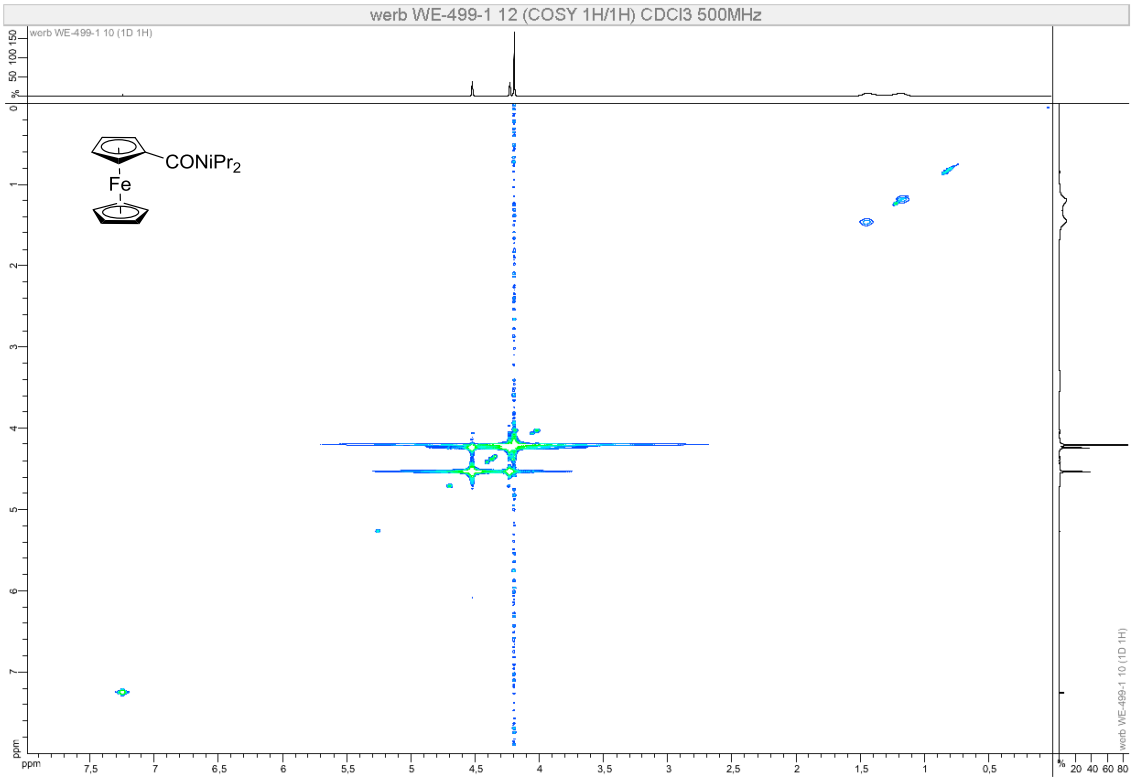
69,984
49,682
46,376
21,349
ppm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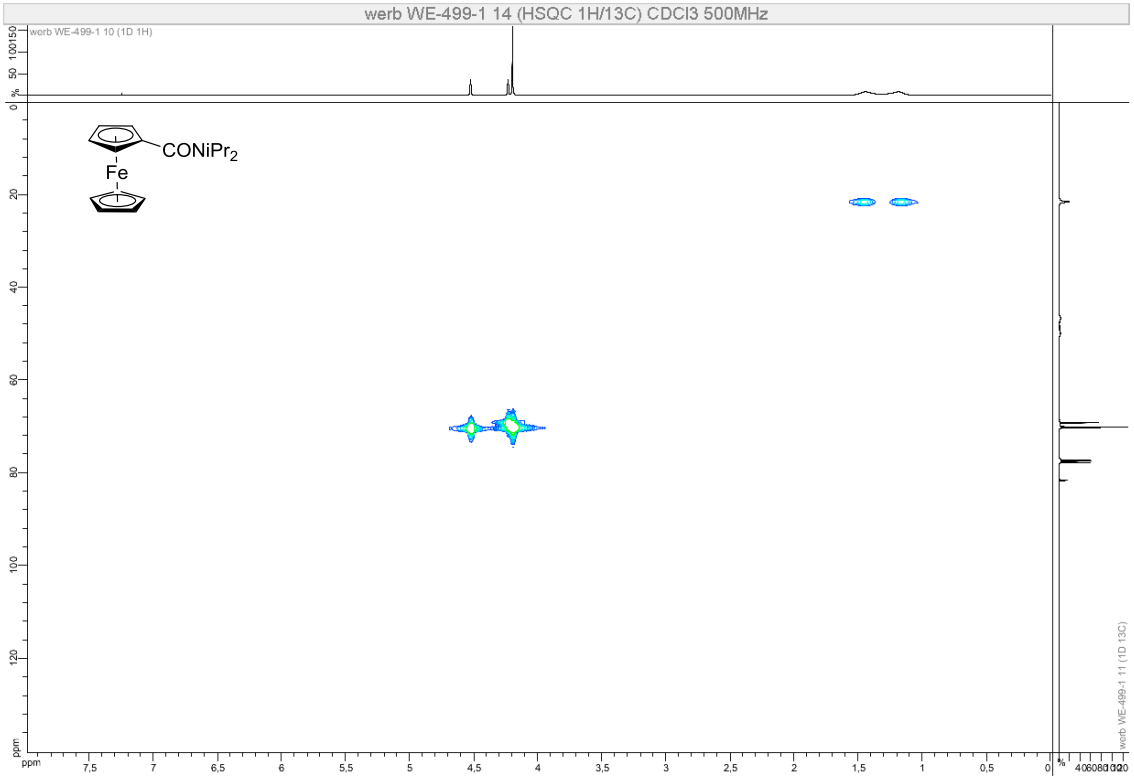
werb WE-499-1 10 (1D 1H)
CONiPr2
Fe
ppm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CONiPr2
Fe
ppm
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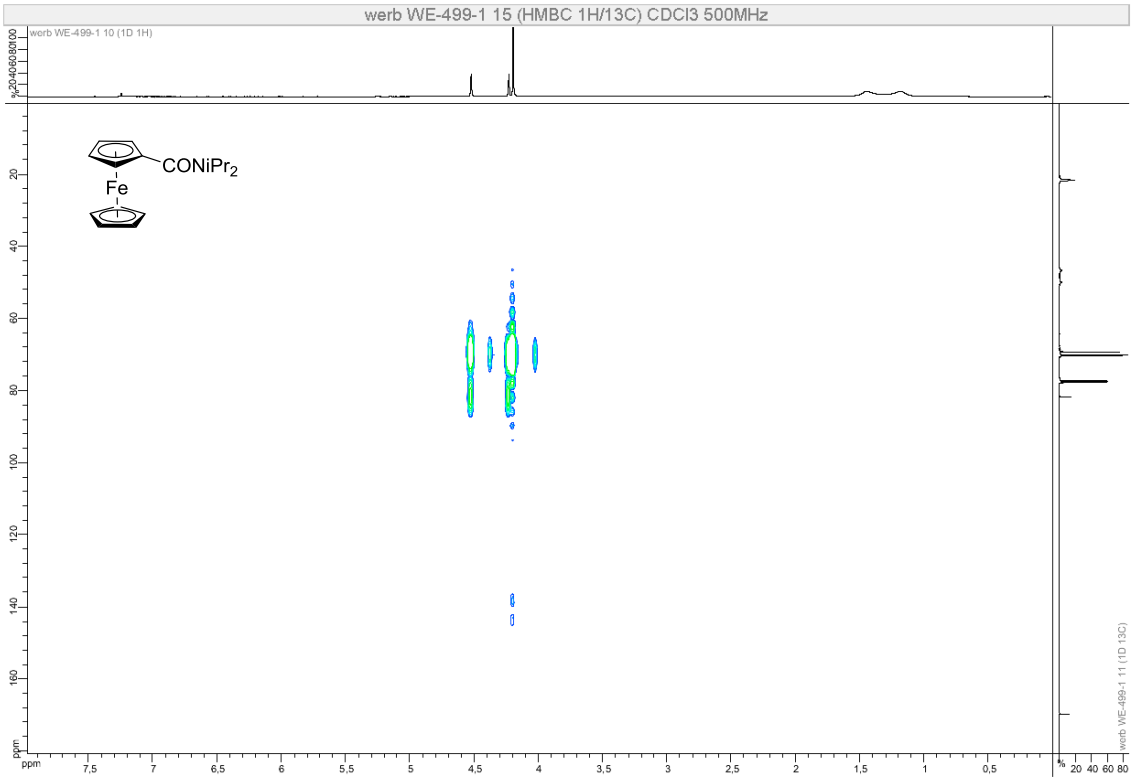
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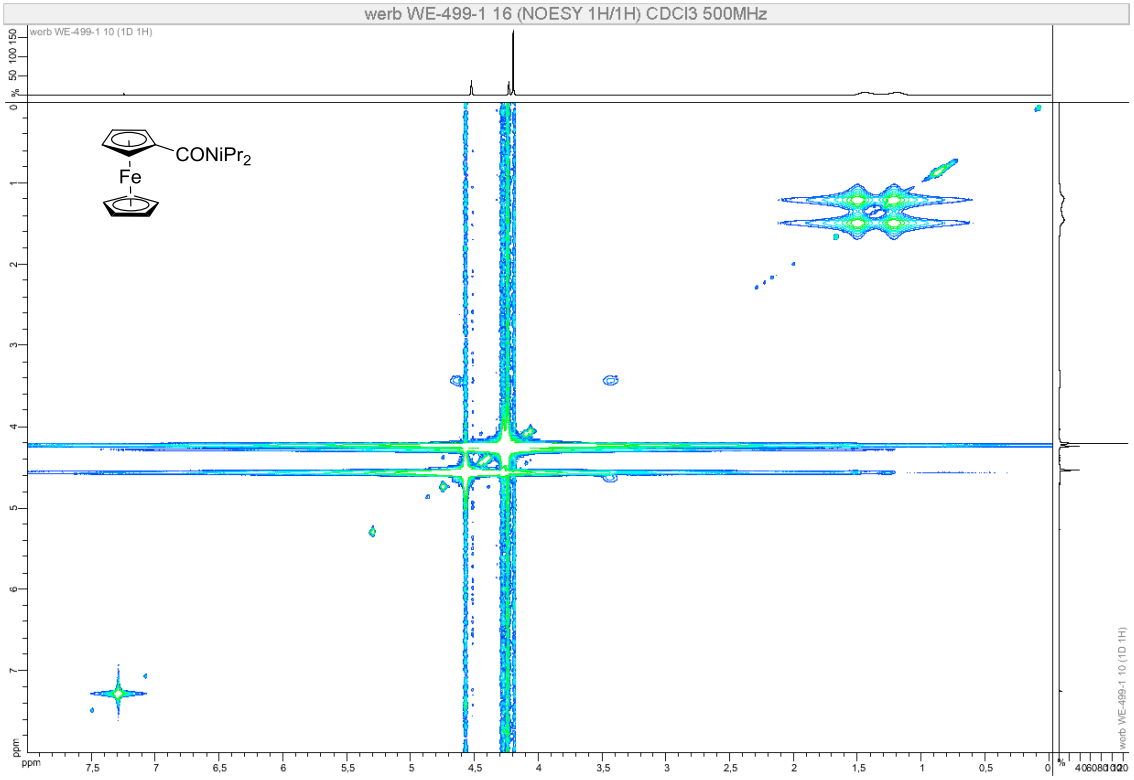
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werb WE-499-1 10 (1D 1H)
CONiPr2
Fe
ppm
7,5
7
6,5
6
5,5
5
4,5
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3,5
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2,5
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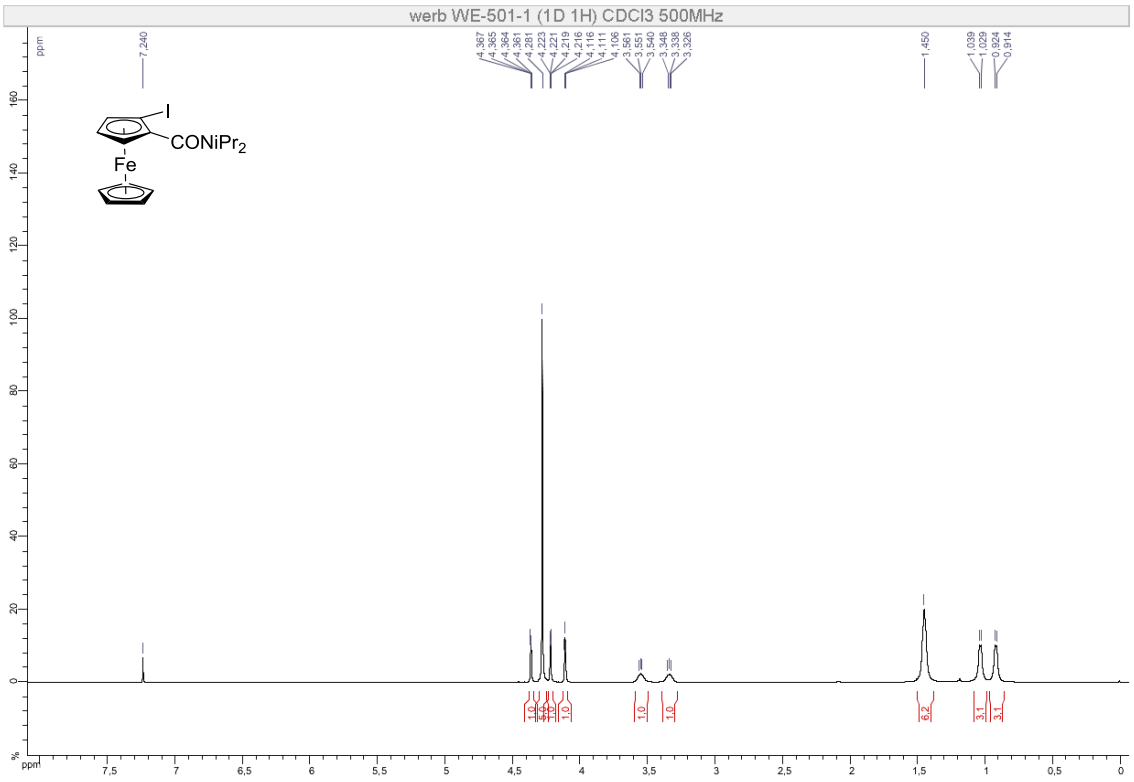
1
0,5
0
werb WE-499-1 10 (1D 1H)

Compound (±)-2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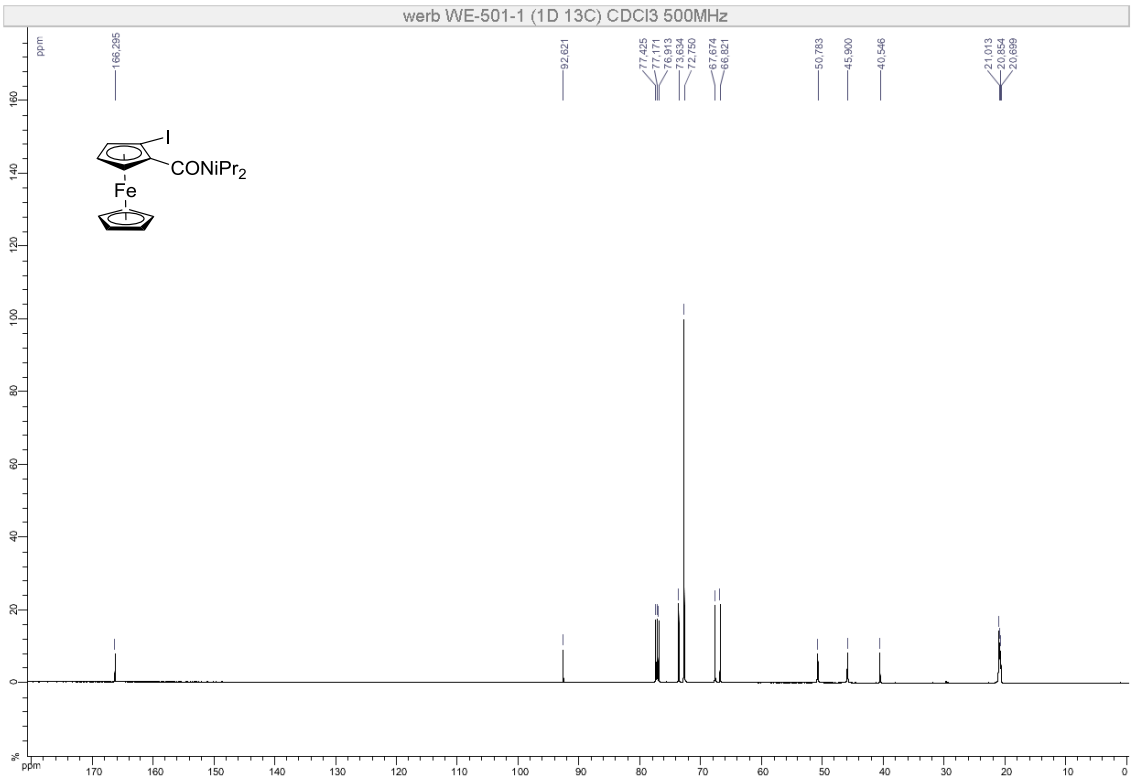
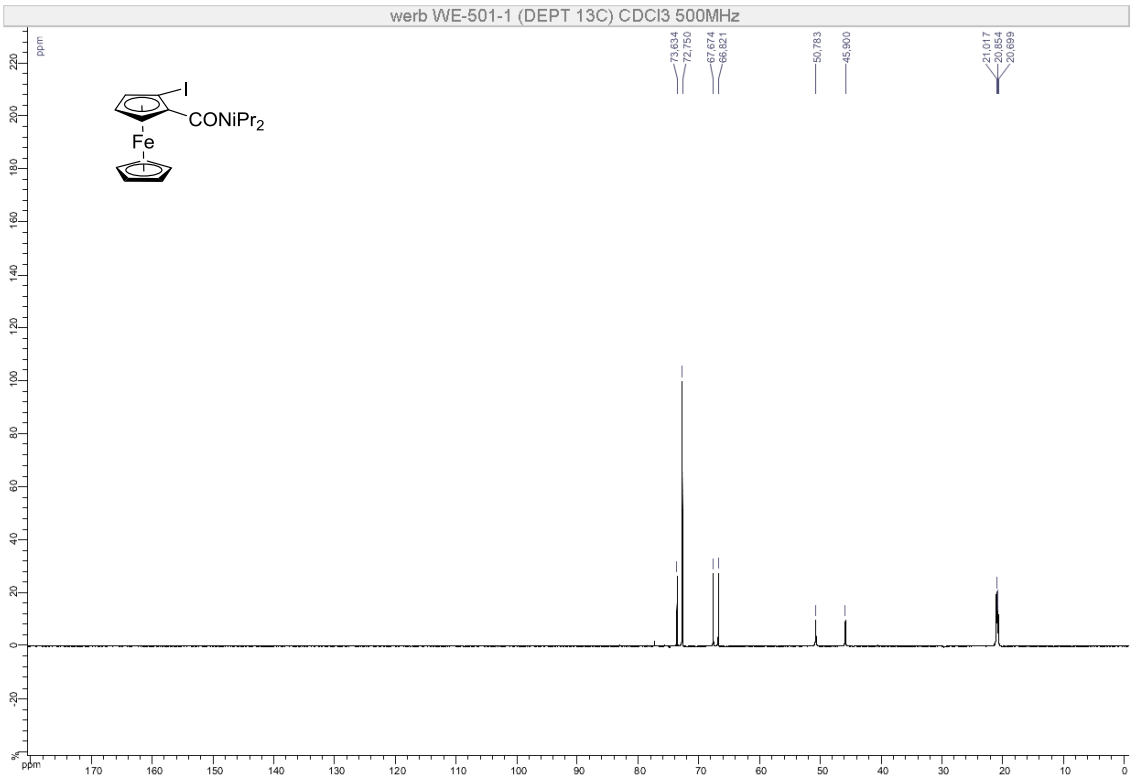
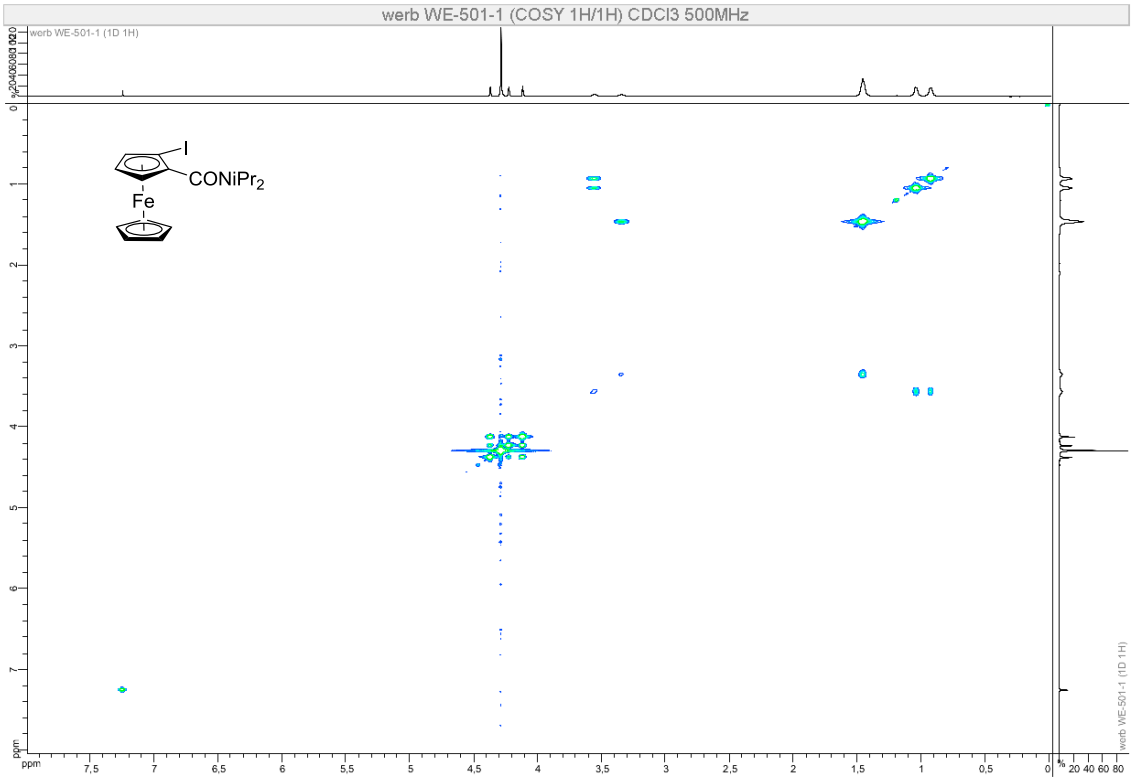
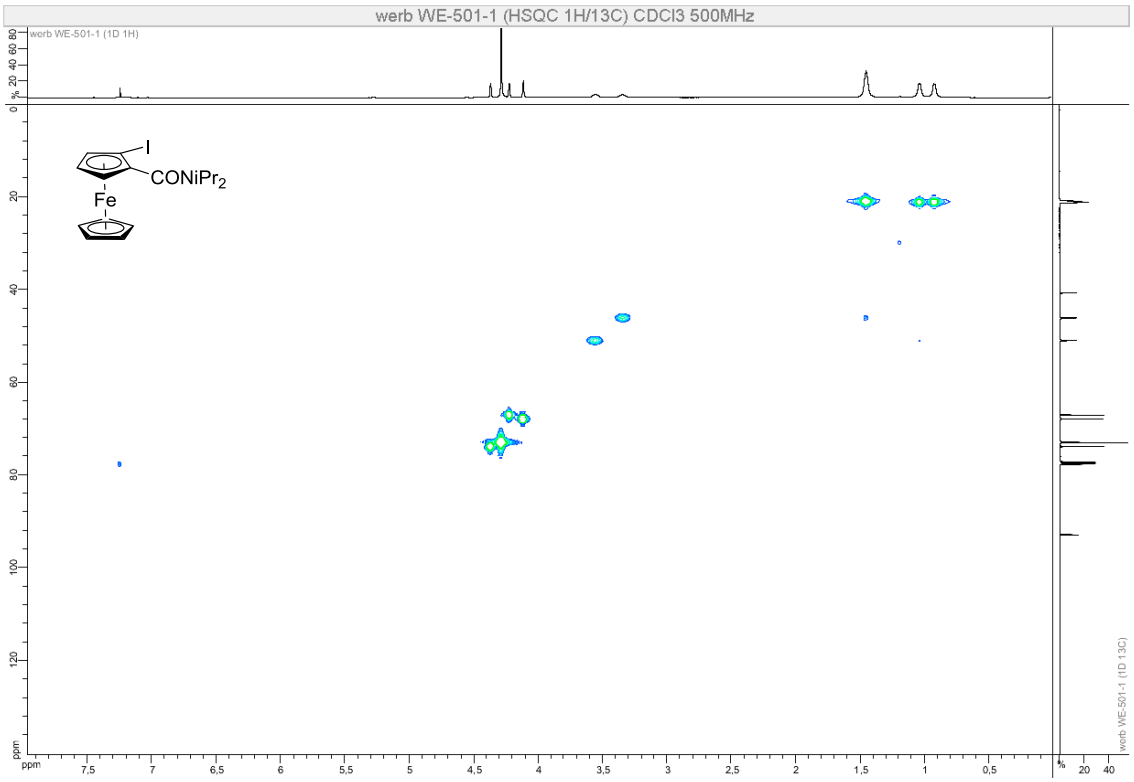
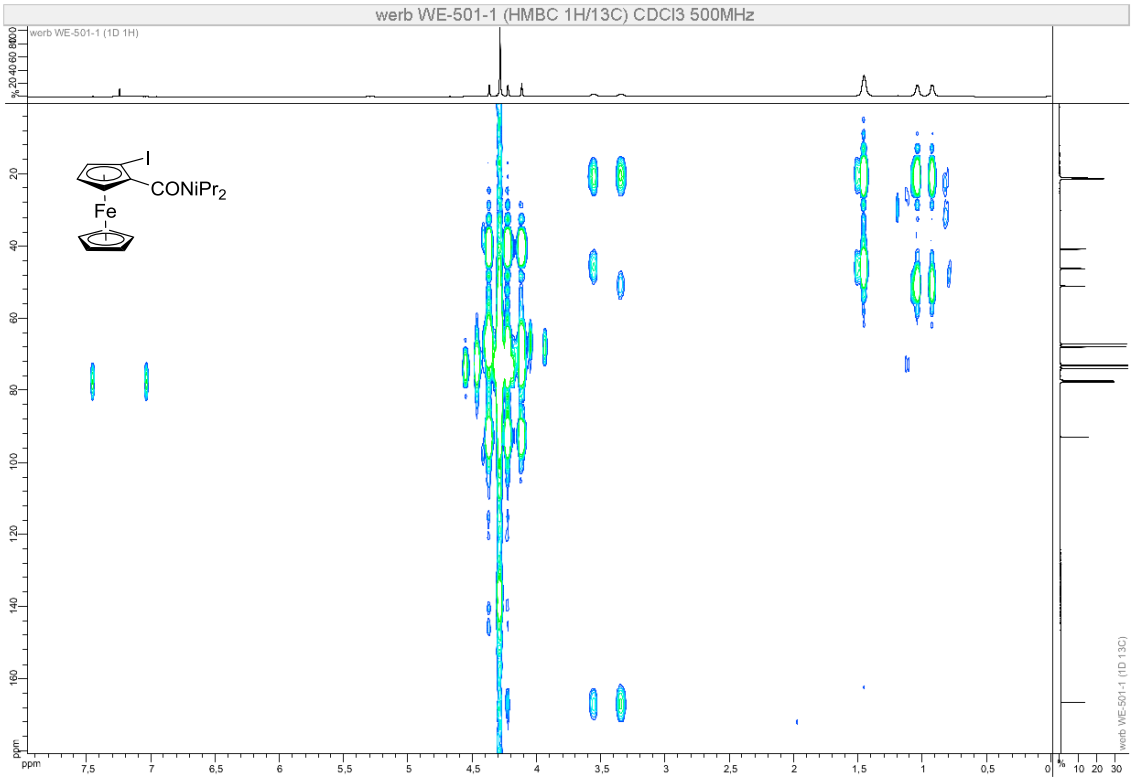
7.240
4.367
4.365
4.364
4.361
4.281
4.223
4.221
4.219
4.216
4.116
4.111
4.106
3.561
3.551
3.540
3.348
3.338
3.326
1.450
1.039
1.029
0.924
0.914
I
CONiPr2
Fe
1.0
1.0
1.0
1.0
1.0
6.2
3.1
3.1
%
ppm
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5
0

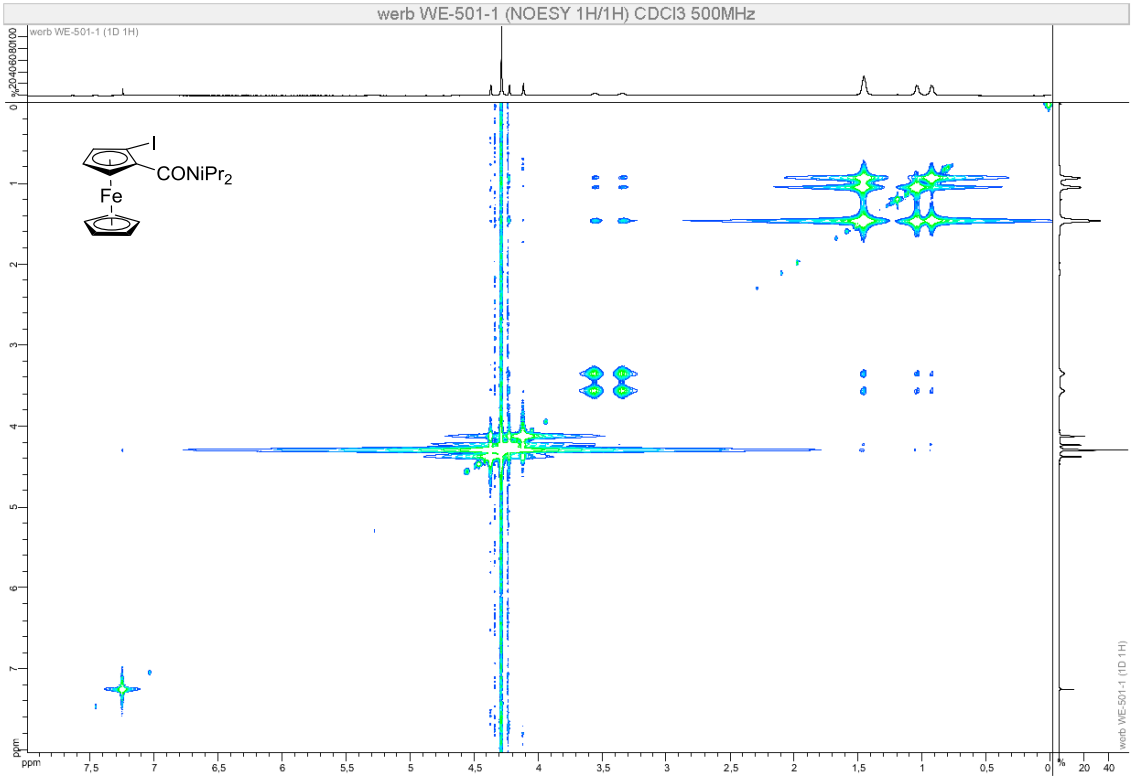

werb WE-501-1 (1D 13C) CDCl3 500MHz
ppm
166.295
92.621
77.425
77.171
76.913
73.634
72.750
67.674
66.821
50.783
45.900
40.546
21.013
20.854
20.699
I
CONiPr2
Fe
%
ppm
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


werb WE-501-1 (DEPT 13C) CDCl3 500MHz
ppm
73,634
72,750
67,674
66,821
50,783
45,900
21,017
20,854
20,699
I
CONiPr2
Fe

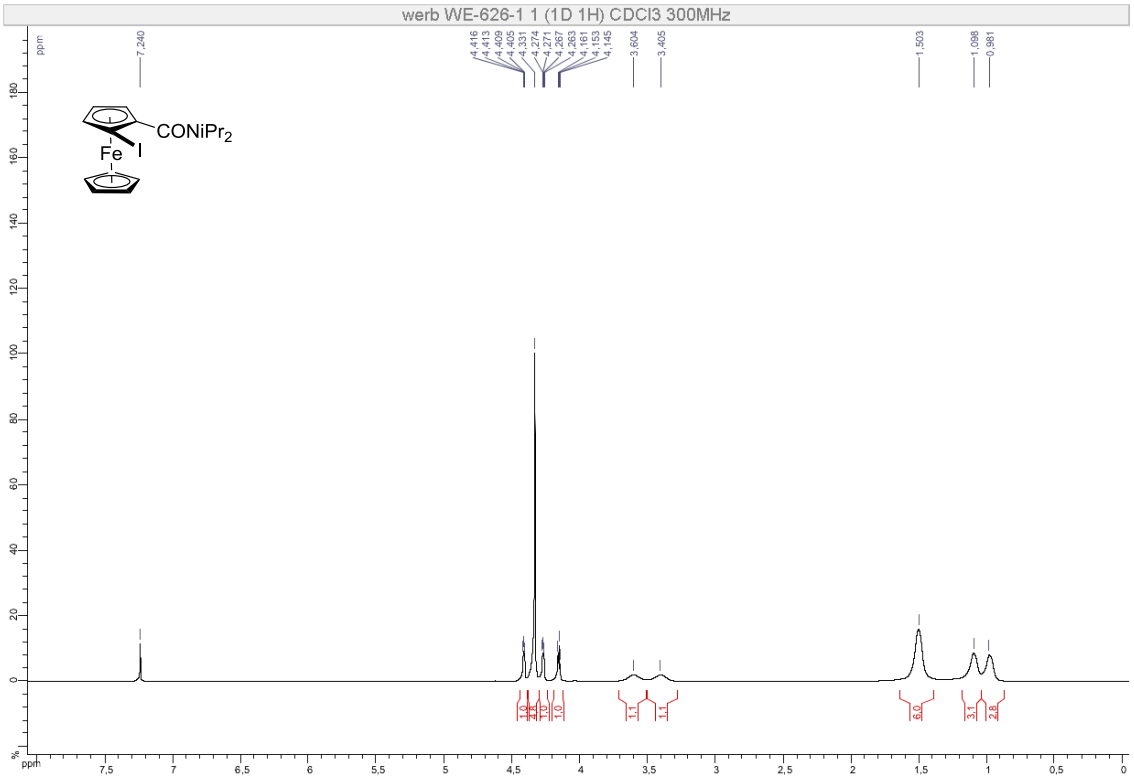

werb WE-501-1 (COSY 1H/1H) CDCl3 500MHz
werb WE-501-1 (1D 1H)
I
CONiPr2
Fe
ppm


werb WE-501-1 (HSQC 1H/13C) CDCl3 500MHz
werb WE-501-1 (1D 1H)
I
CONiPr2
Fe
ppm
werb WE-501-1 (1D 13C)

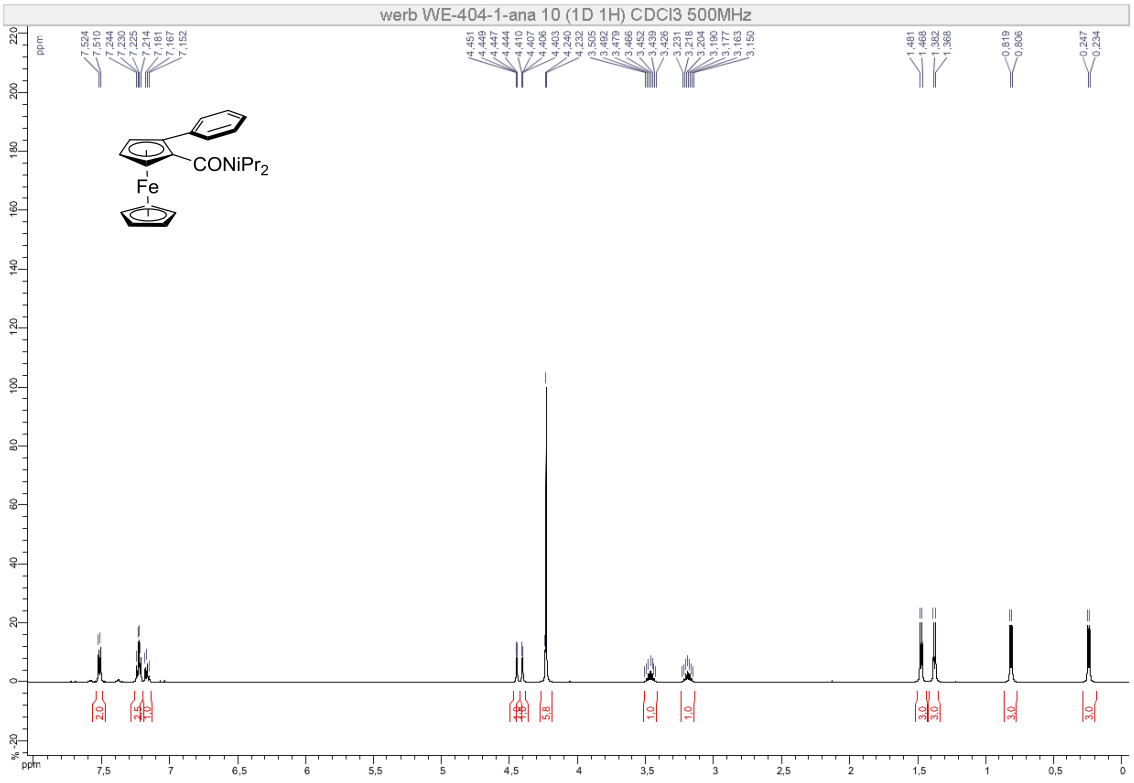

werb WE-501-1 (HMBC 1H/13C) CDCl3 500MHz
werb WE-501-1 (1D 1H)
I
CONiPr2
Fe
ppm
werb WE-501-1 (1D 13C)

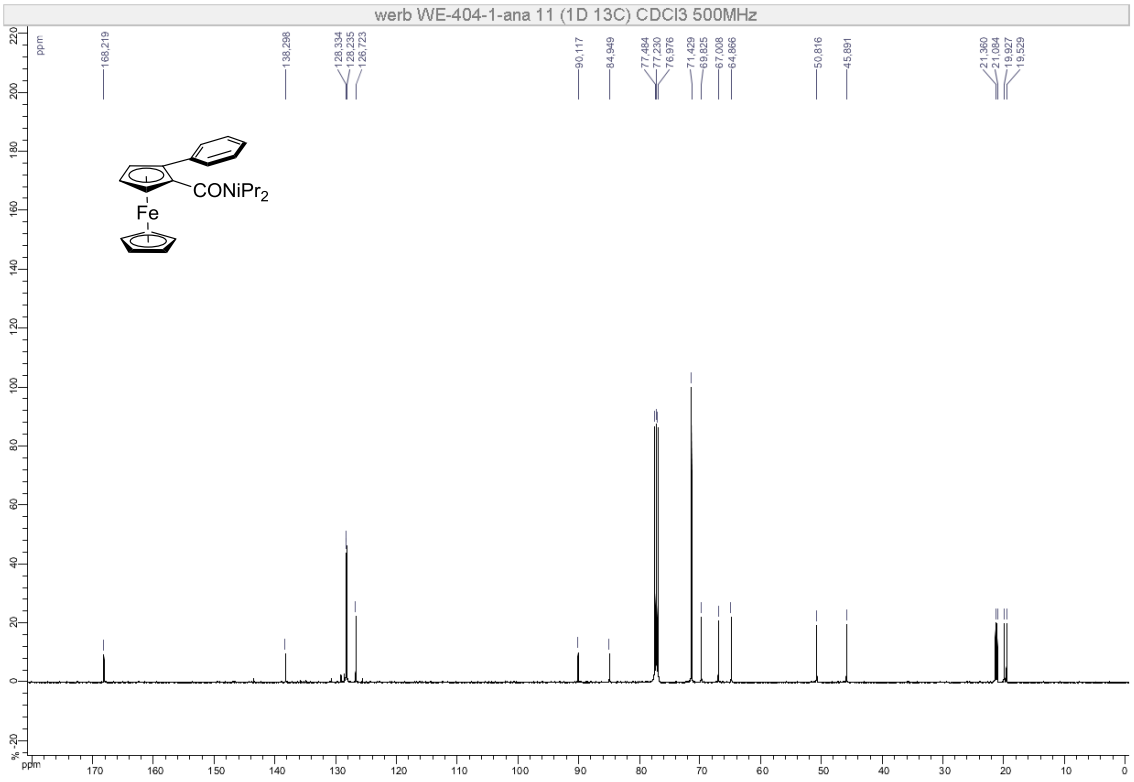


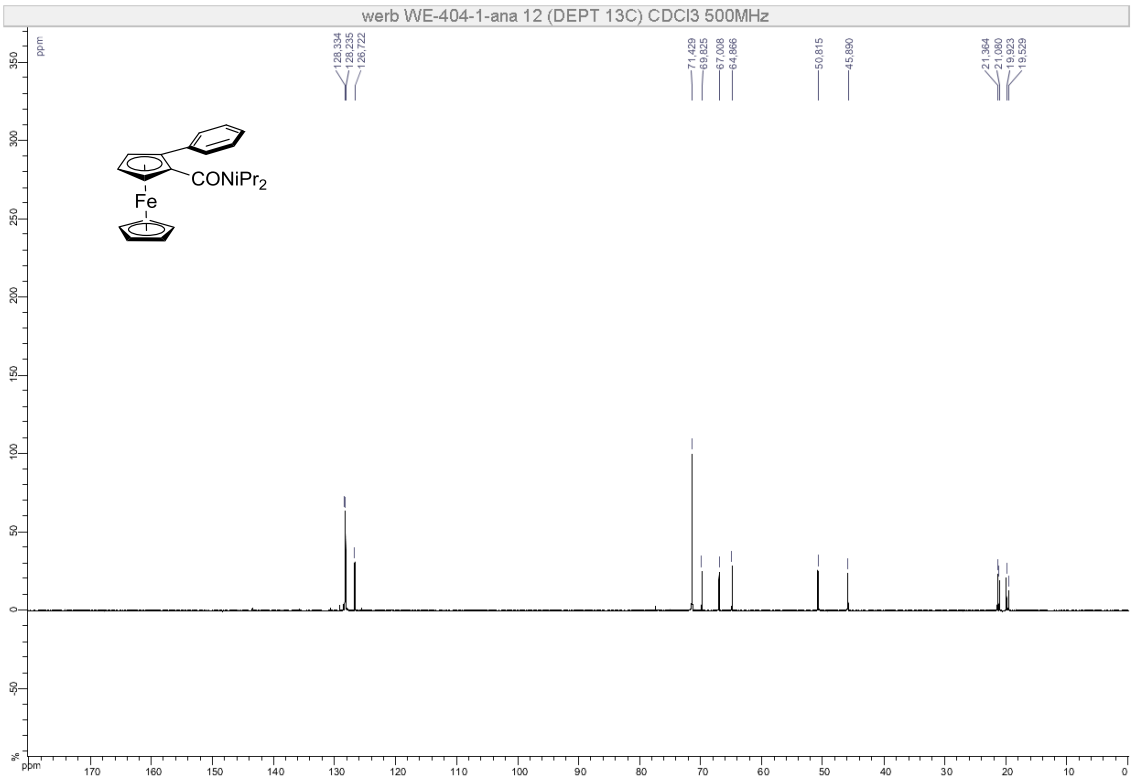
Compound (S_p)-2

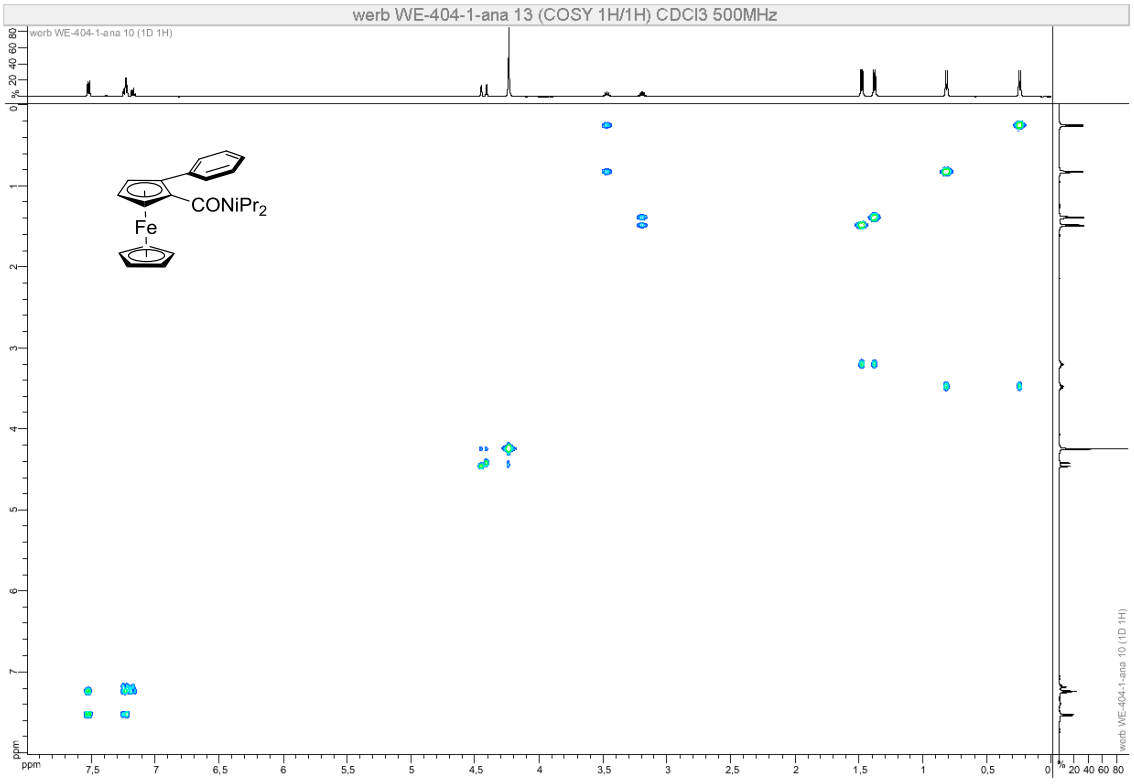


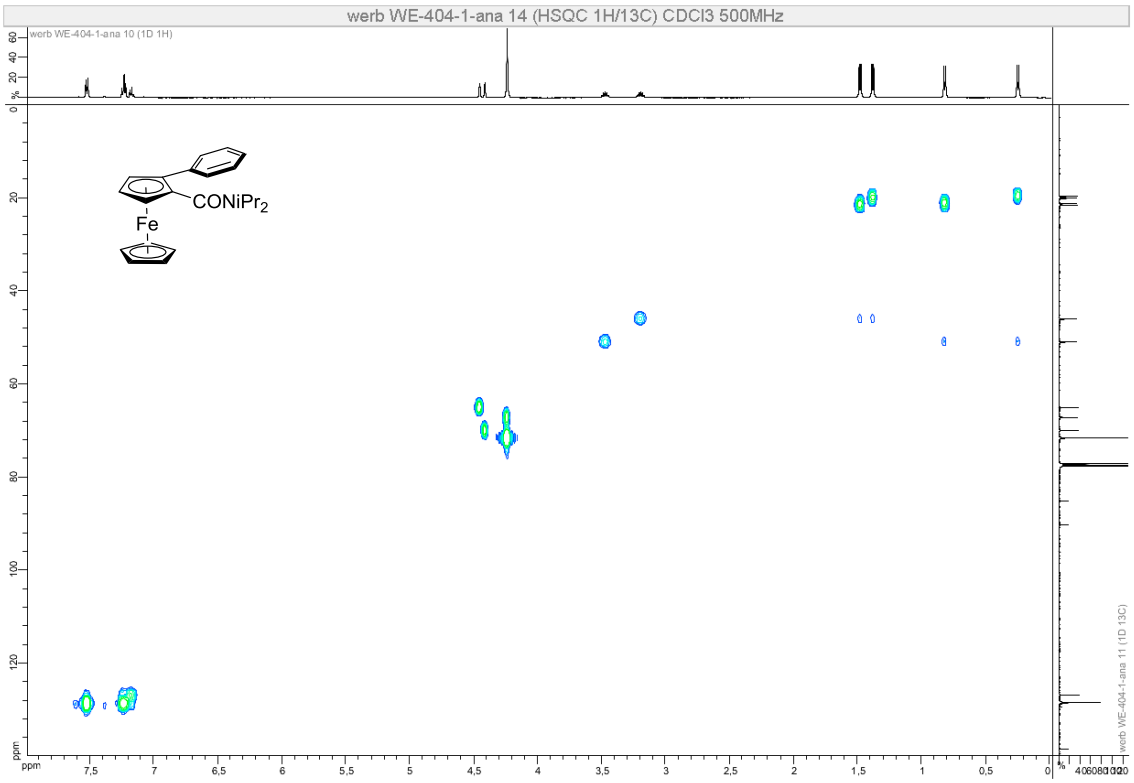
Compound (±)-4

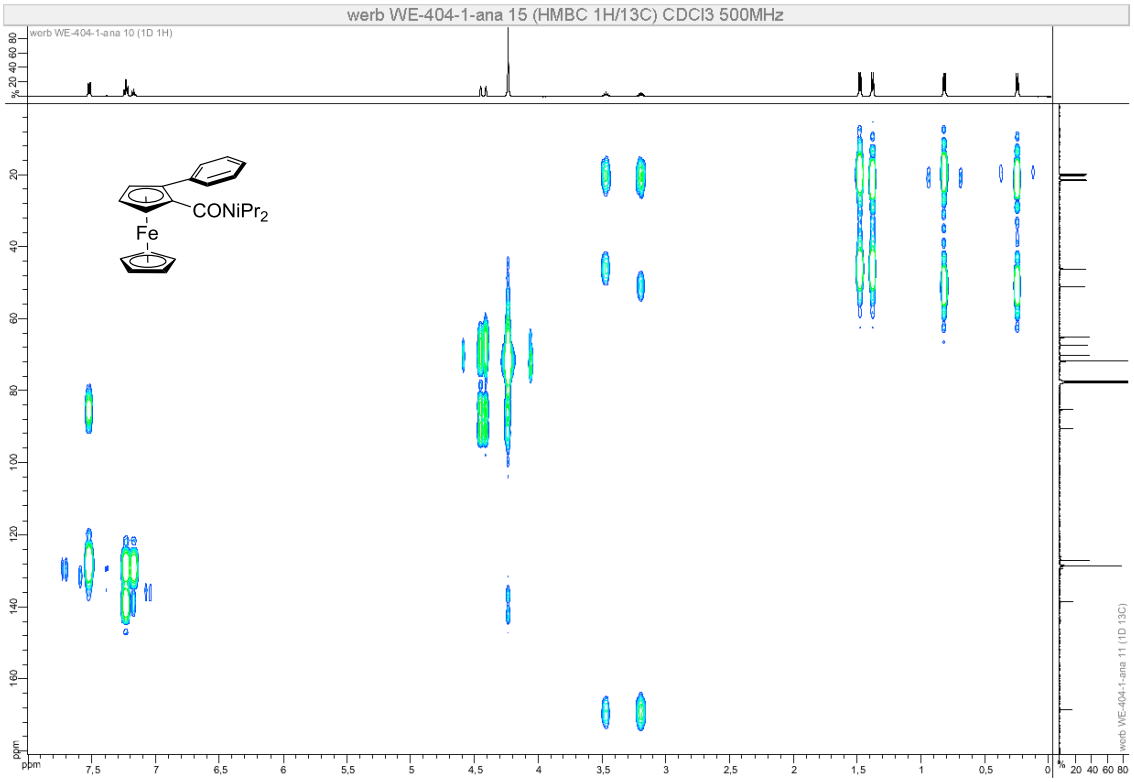

werb WE-404-1-ana 10 (1D 1H) CDCl3 500MHz
CONiPr2
Fe

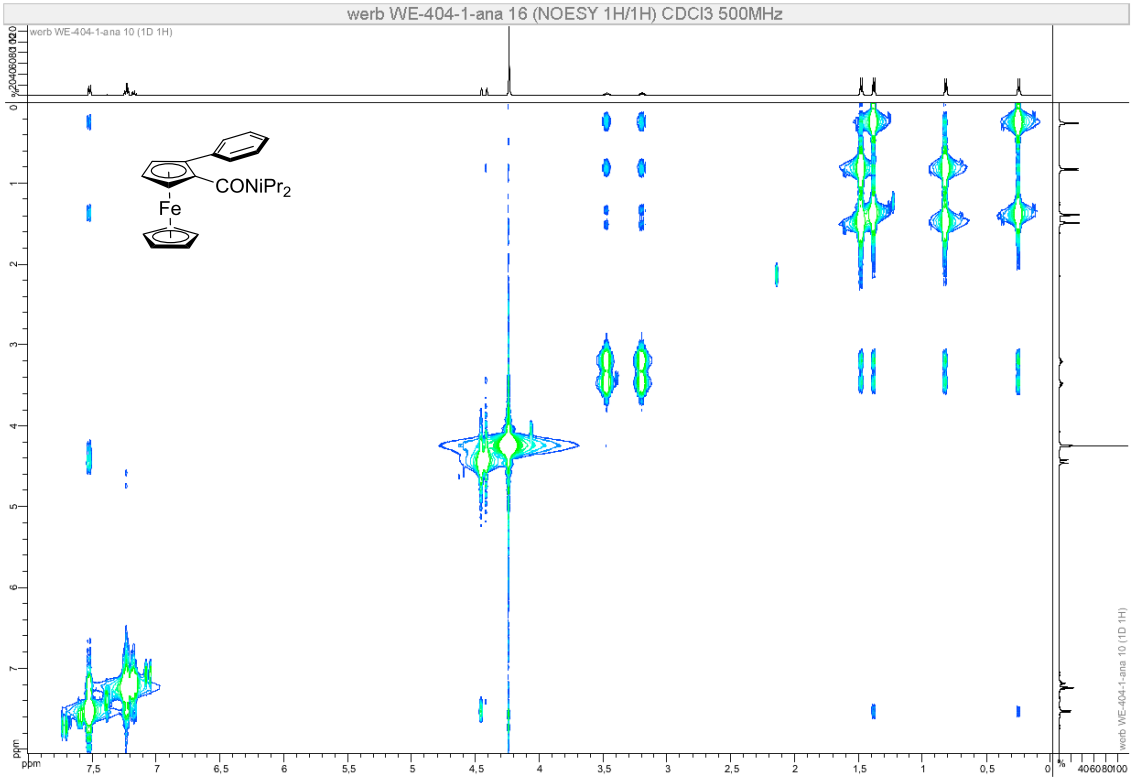

werb WE-404-1-ana 11 (1D 13C) CDCl3 500MHz
CONiPr2
Fe


werb WE-404-1-ana 12 (DEPT 13C) CDCl3 500MHz
128,334
128,235
126,722
71,429
69,825
67,008
64,866
50,815
45,890
21,364
21,080
19,923
19,529
CONiPr2
Fe

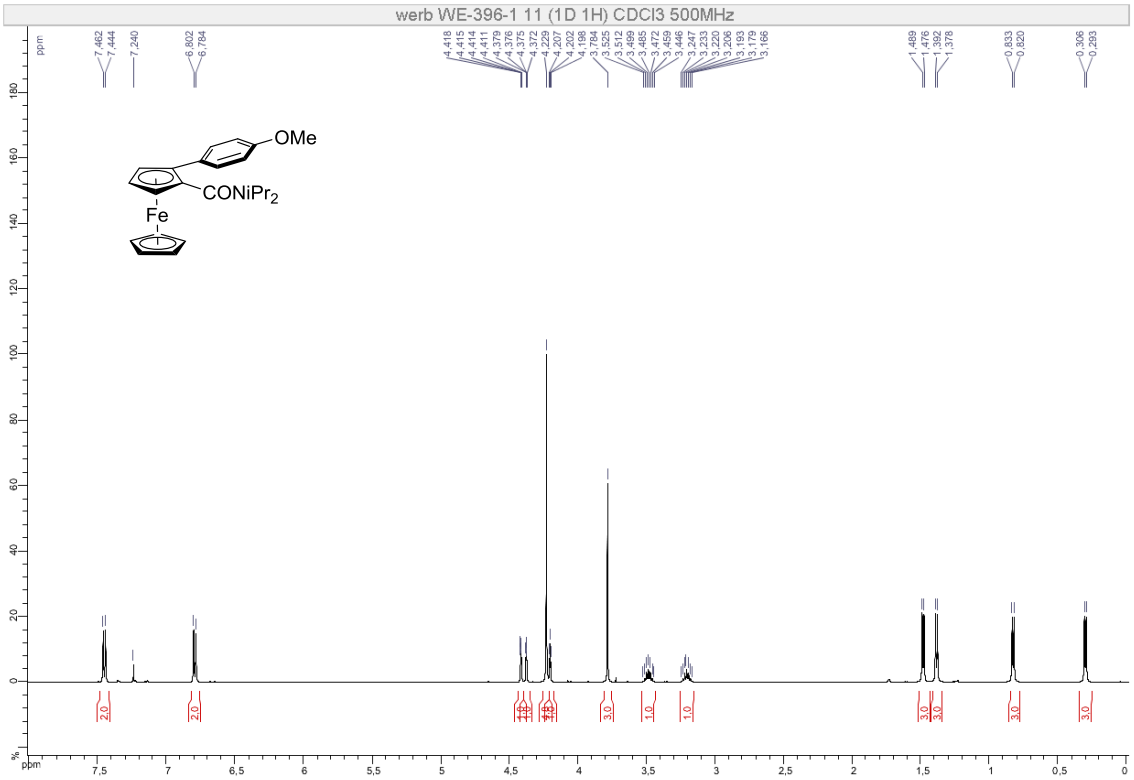

werb WE-404-1-ana 13 (COSY 1H/1H) CDCl3 500MHz
werb WE-404-1-ana 10 (1D 1H)
CONiPr2
Fe

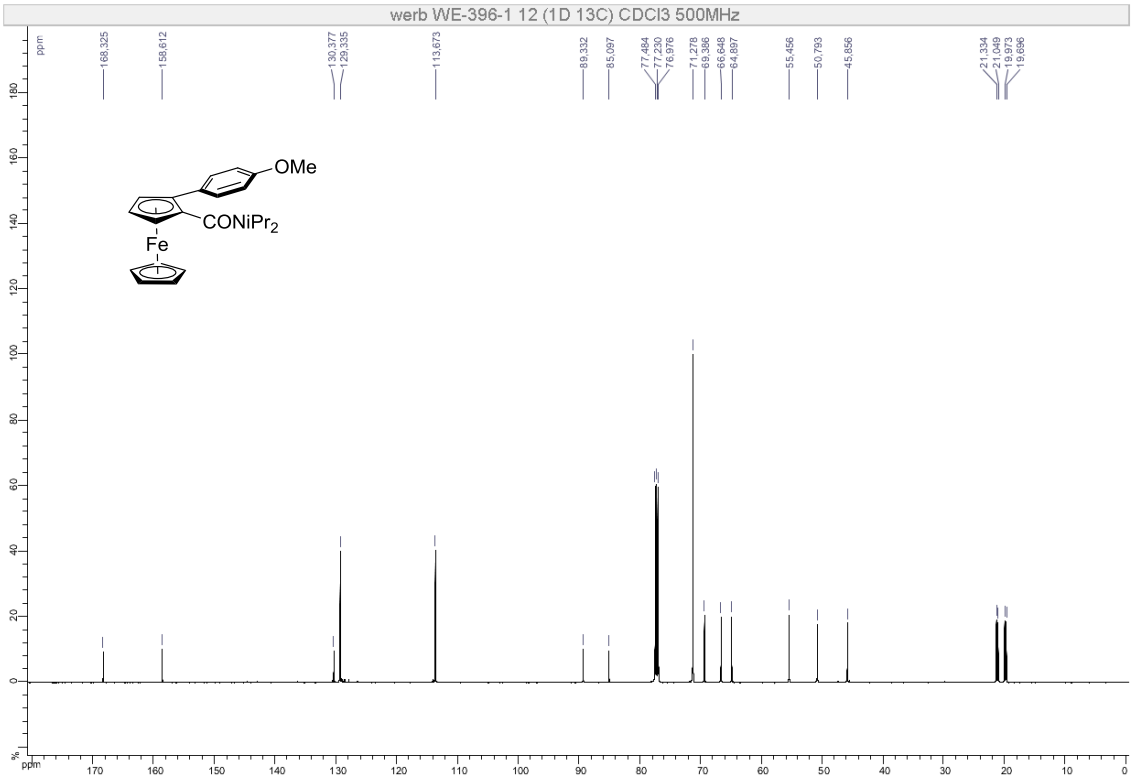

werb WE-404-1-ana 14 (HSQC 1H/13C) CDCl3 500MHz
werb WE-404-1-ana 10 (1D 1H)
CONiPr2
Fe
ppm
werb WE-404-1-ana 11 (1D 13C)

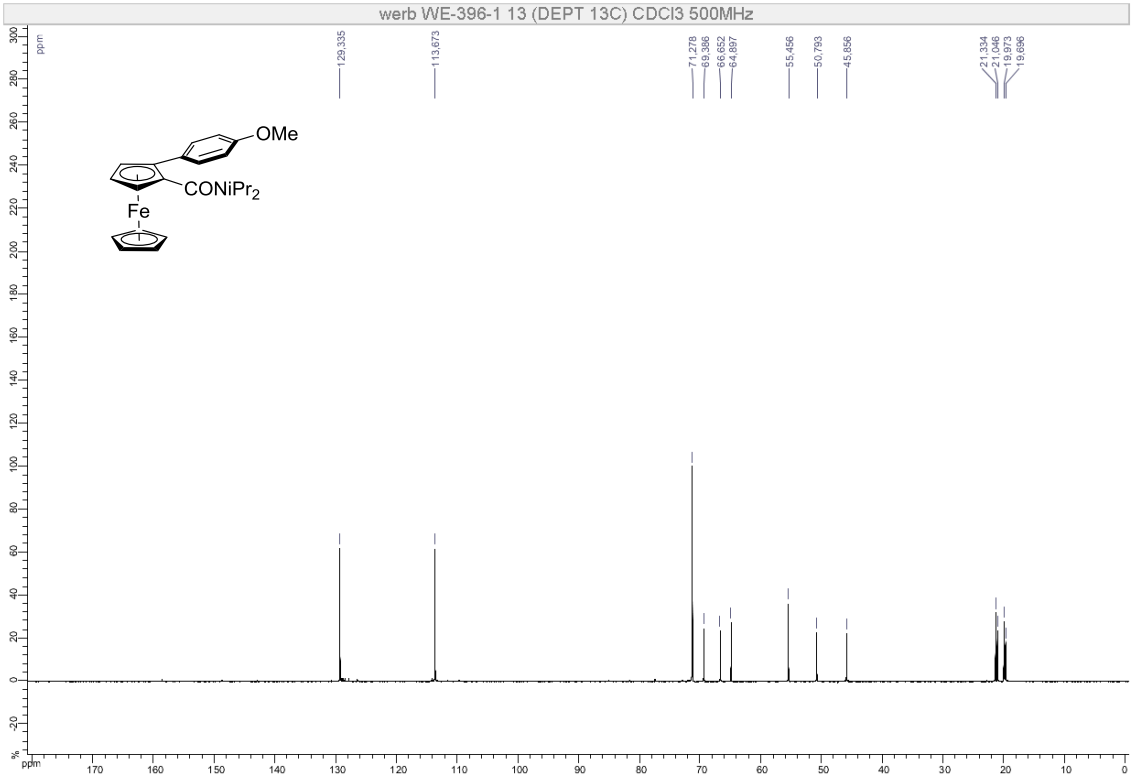

werb WE-404-1-ana 15 (HMBC 1H/13C) CDCl3 500MHz
werb WE-404-1-ana 10 (1D 1H)
CONiPr2
Fe
ppm
werb WE-404-1-ana 11 (1D 13C)

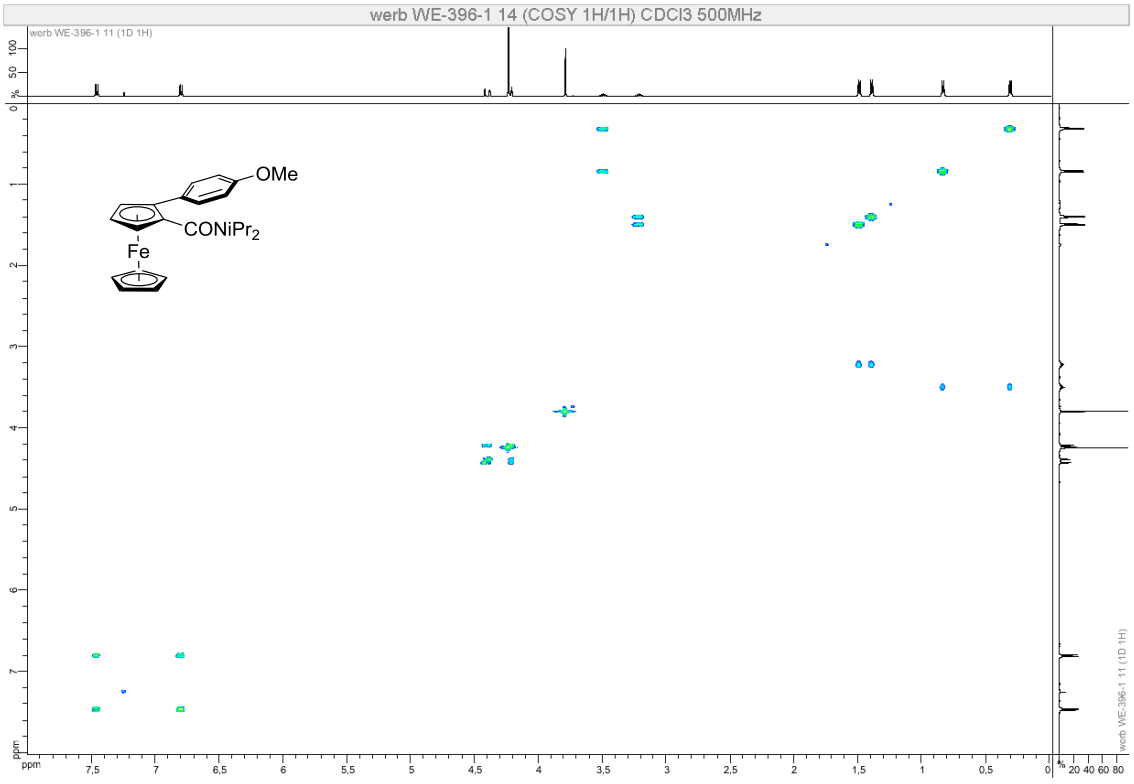

werb WE-404-1-ana 16 (NOESY 1H/1H) CDCl3 500MHz
werb WE-404-1-ana 10 (1D 1H)
CONiPr2
Fe
ppm
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5
0
40
60
80
100
werb WE-404-1-ana 10 (1D 1H)

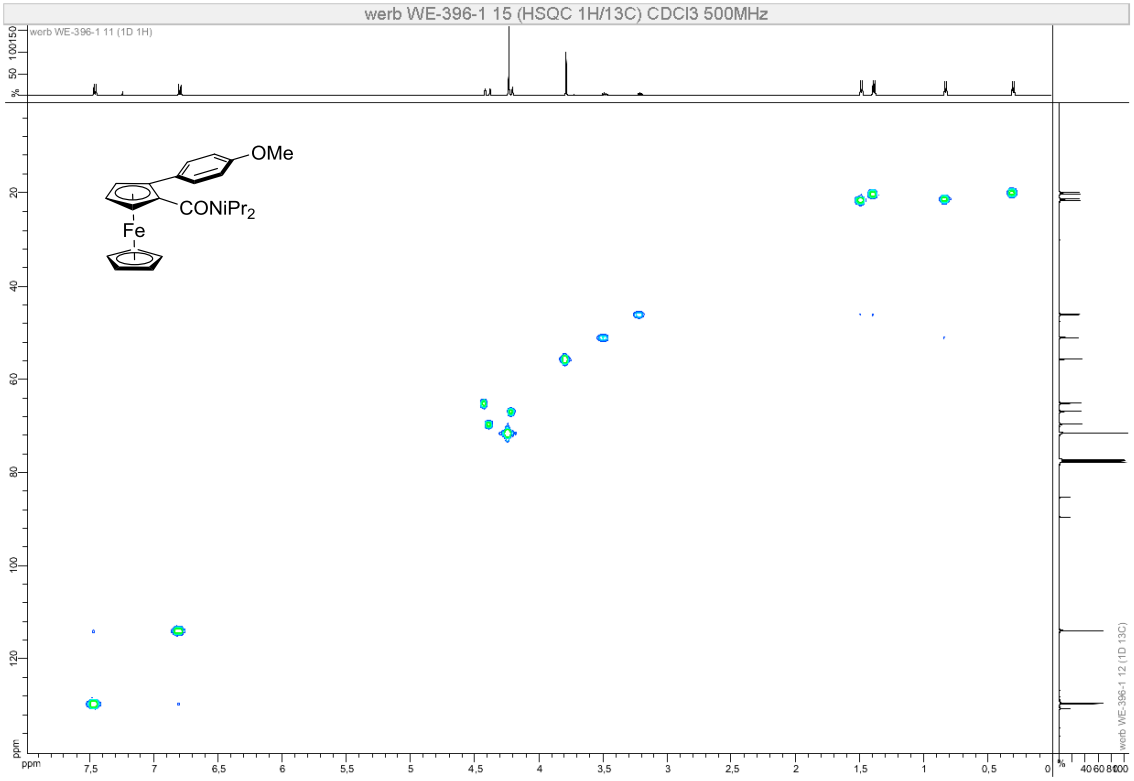
Compound (±)-5

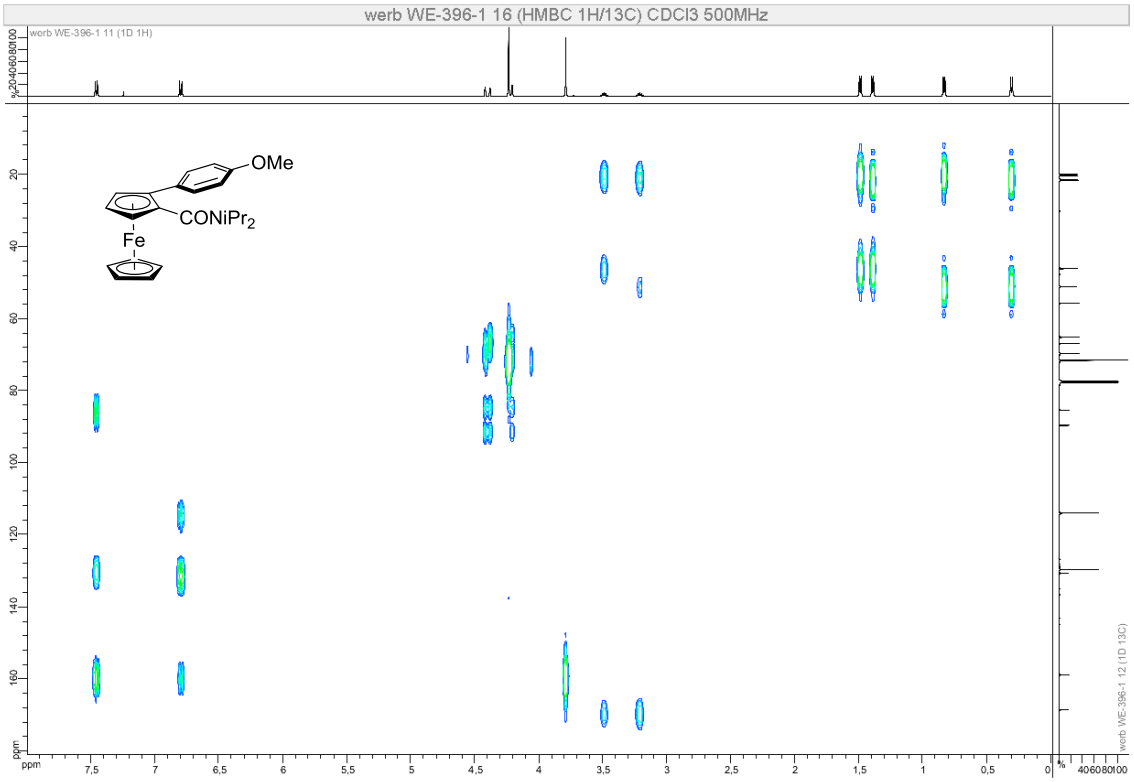

werb WE-396-1 11 (1D 1H) CDCl3 500MHz
OMe
CONiPr2
Fe

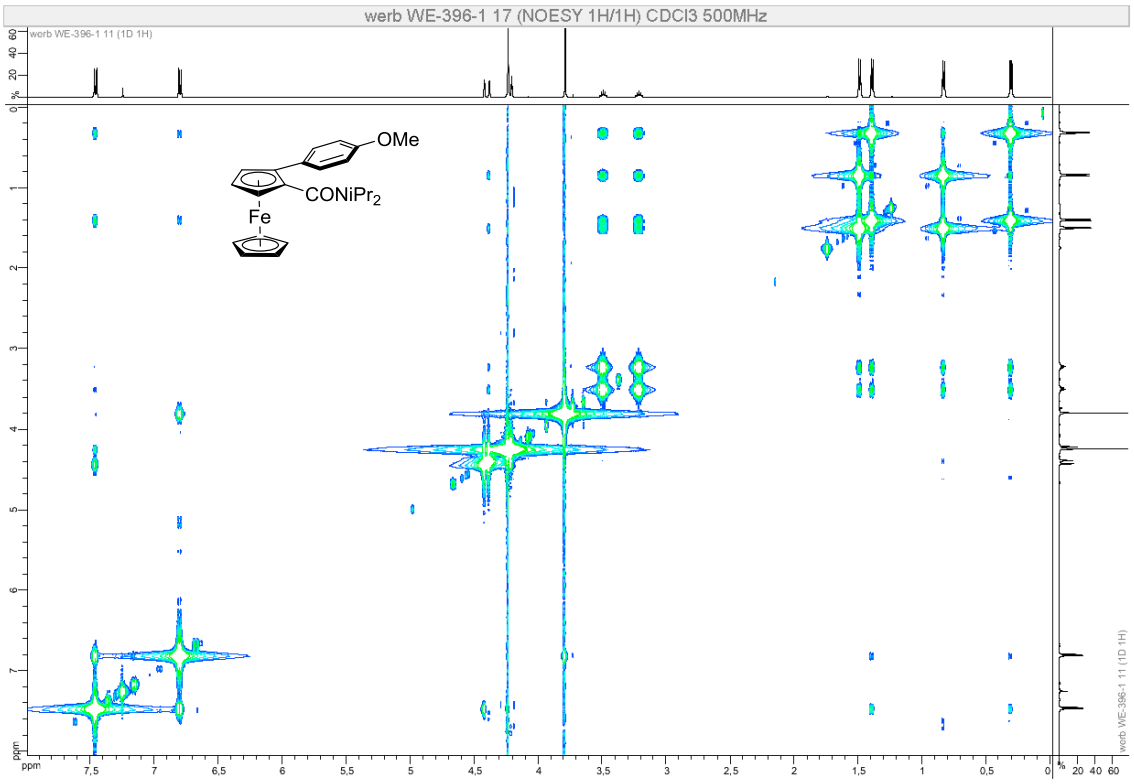

werb WE-396-1 12 (1D 13C) CDCl3 500MHz
OMe
CONiPr2
Fe


werb WE-396-1 13 (DEPT 13C) CDCl3 500MHz
129,335
113,673
71,278
69,386
66,652
64,897
55,456
50,793
45,856
21,334
21,046
19,973
19,696
OMe
CONiPr2
Fe
ppm

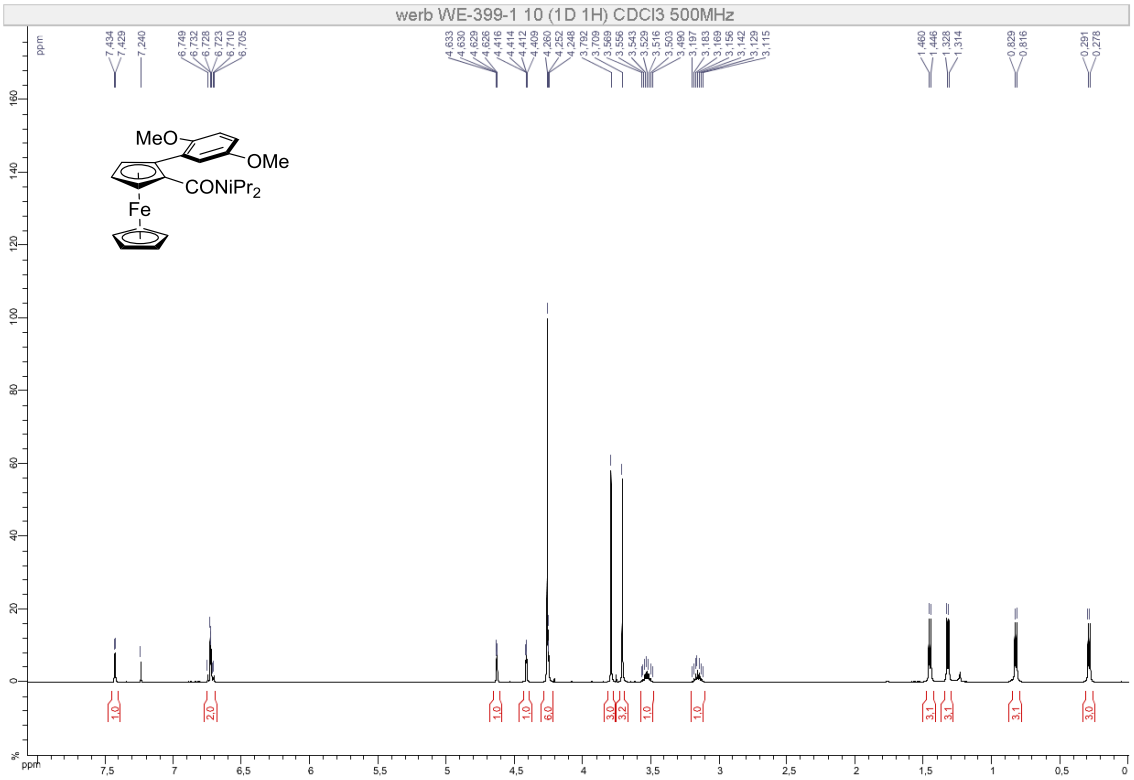

werb WE-396-1 14 (COSY 1H/1H) CDCl3 500MHz
werb WE-396-1 11 (1D 1H)
OMe
CONiPr2
Fe
ppm

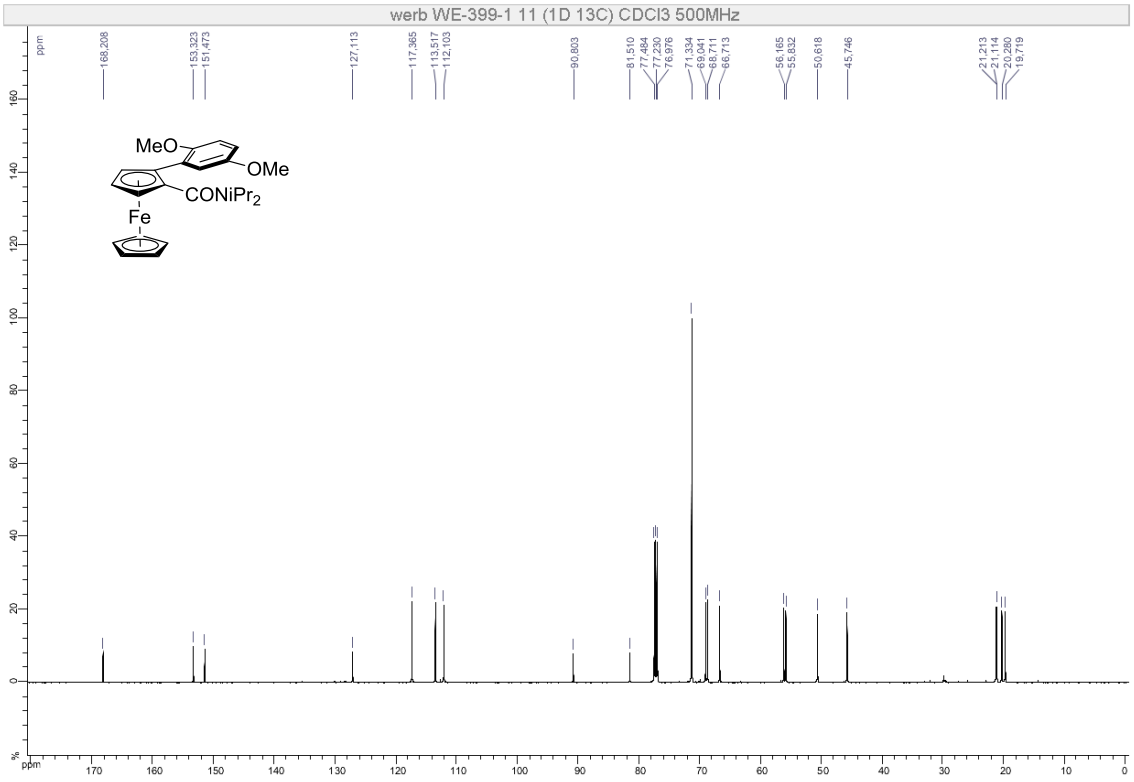

werb WE-396-1 15 (HSQC 1H/13C) CDCl3 500MHz
werb WE-396-1 11 (1D 1H)
OMe
CONiPr2
Fe
ppm
werb WE-396-1 12 (1D 13C)

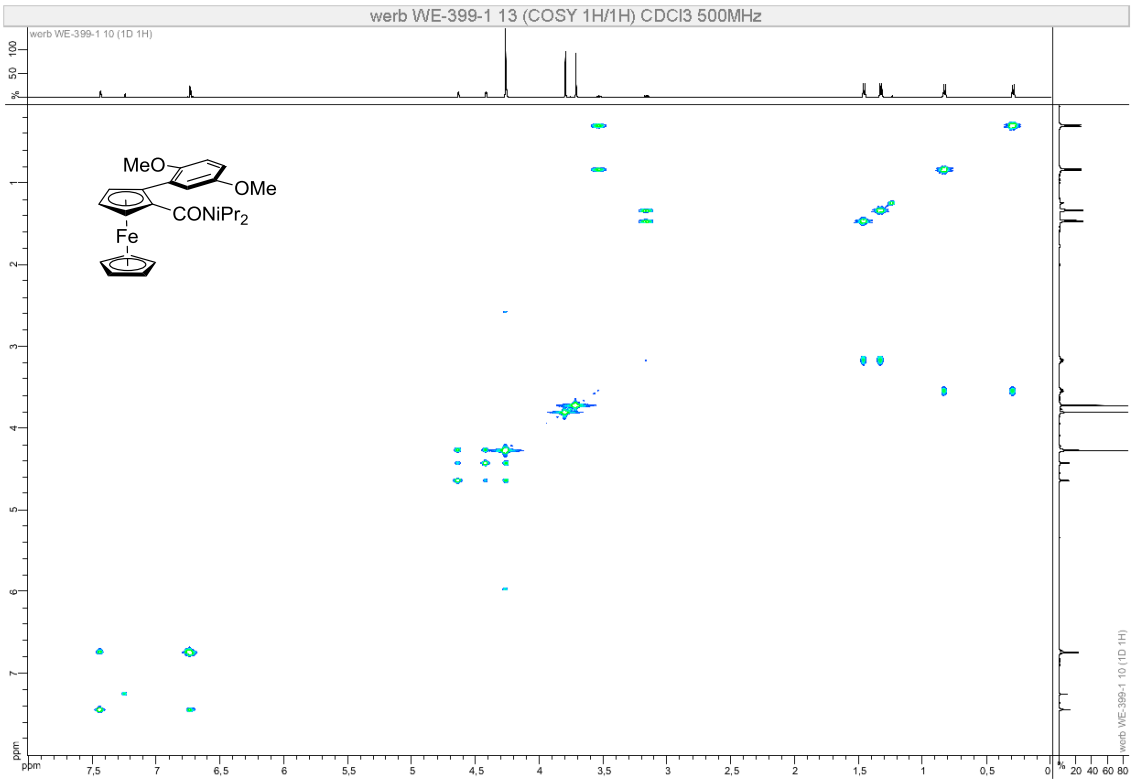

werb WE-396-1 16 (HMBC 1H/13C) CDCl3 500MHz
werb WE-396-1 11 (1D 1H)
OMe
CONiPr2
Fe
ppm
werb WE-396-1 12 (1D 13C)

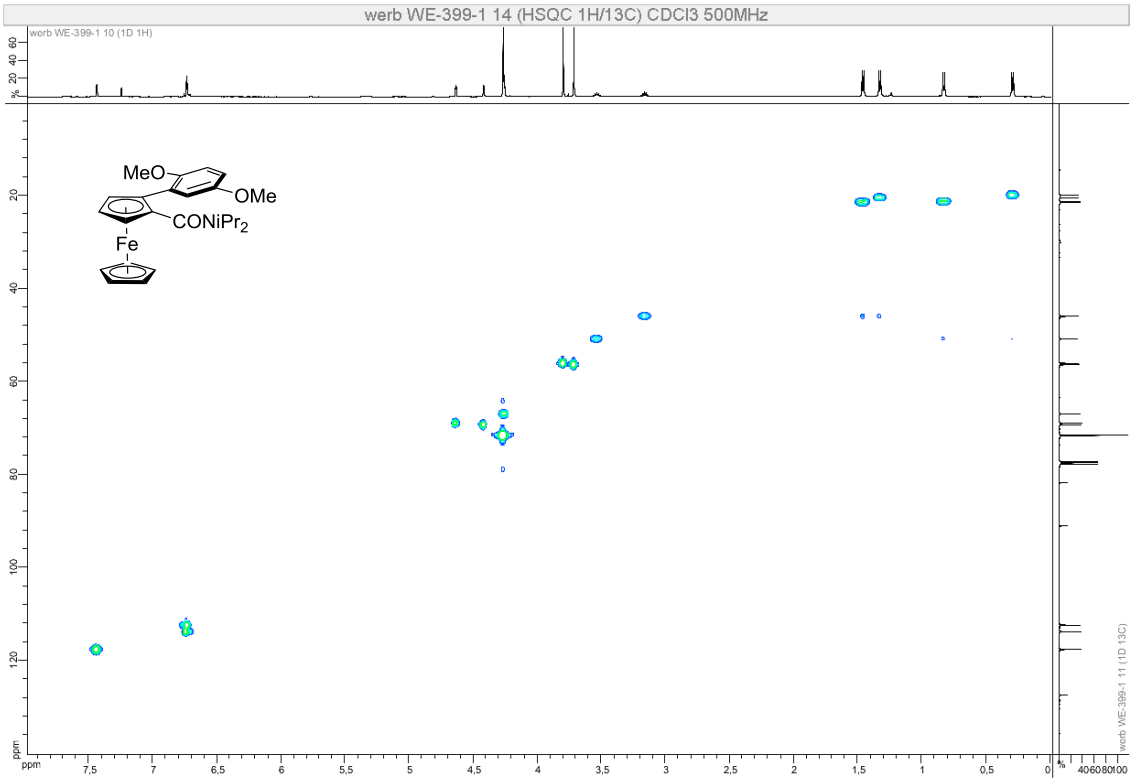

werb WE-396-1 17 (NOESY 1H/1H) CDCl3 500MHz
werb WE-396-1 11 (1D 1H)
OMe
CONiPr2
Fe
ppm
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5
0
20 40 60
werb WE-396-1 11 (1D 1H)

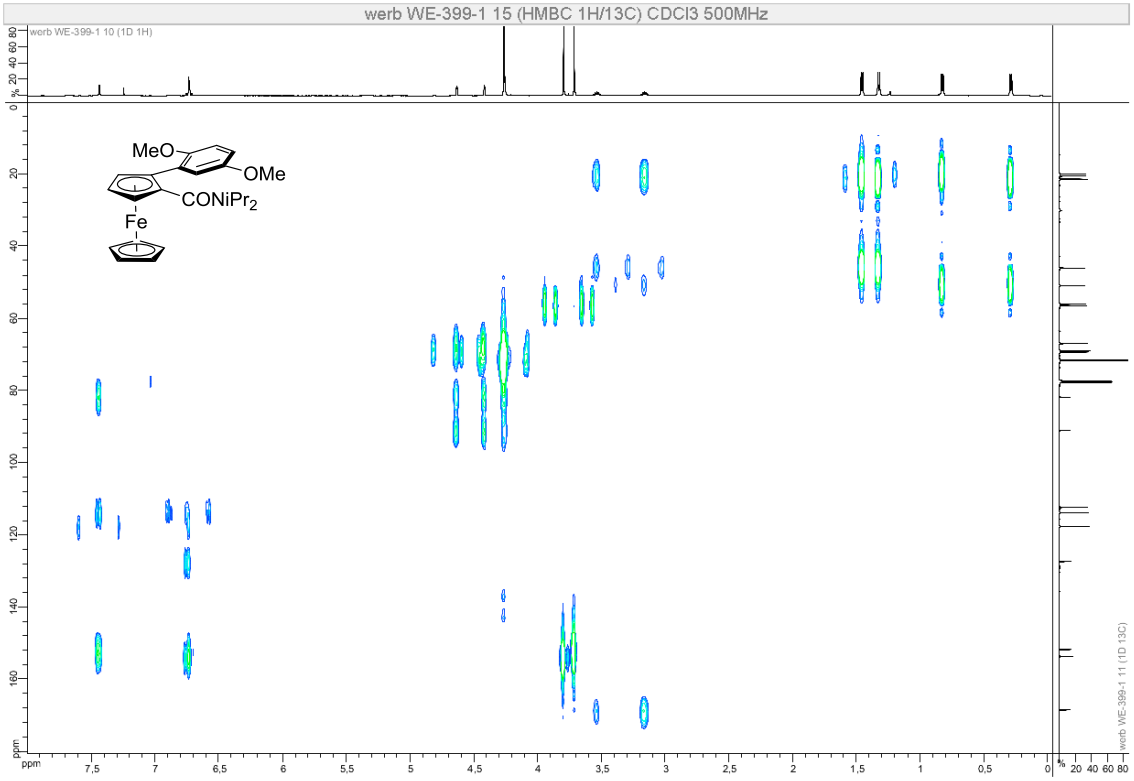
Compound (±)-6

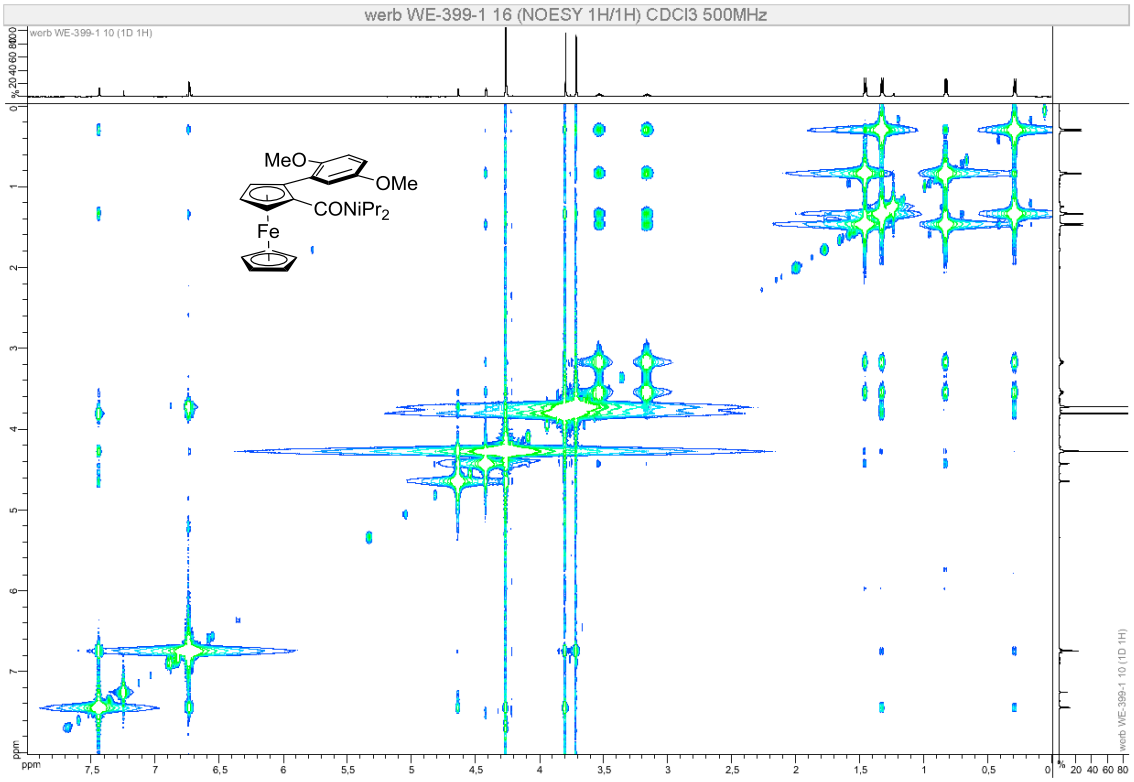

werb WE-399-1 10 (1D 1H) CDCl3 500MHz
MeO
OMe
CONiPr2
Fe


werb WE-399-1 11 (1D 13C) CDCl3 500MHz
MeO
OMe
CONiPr2
Fe

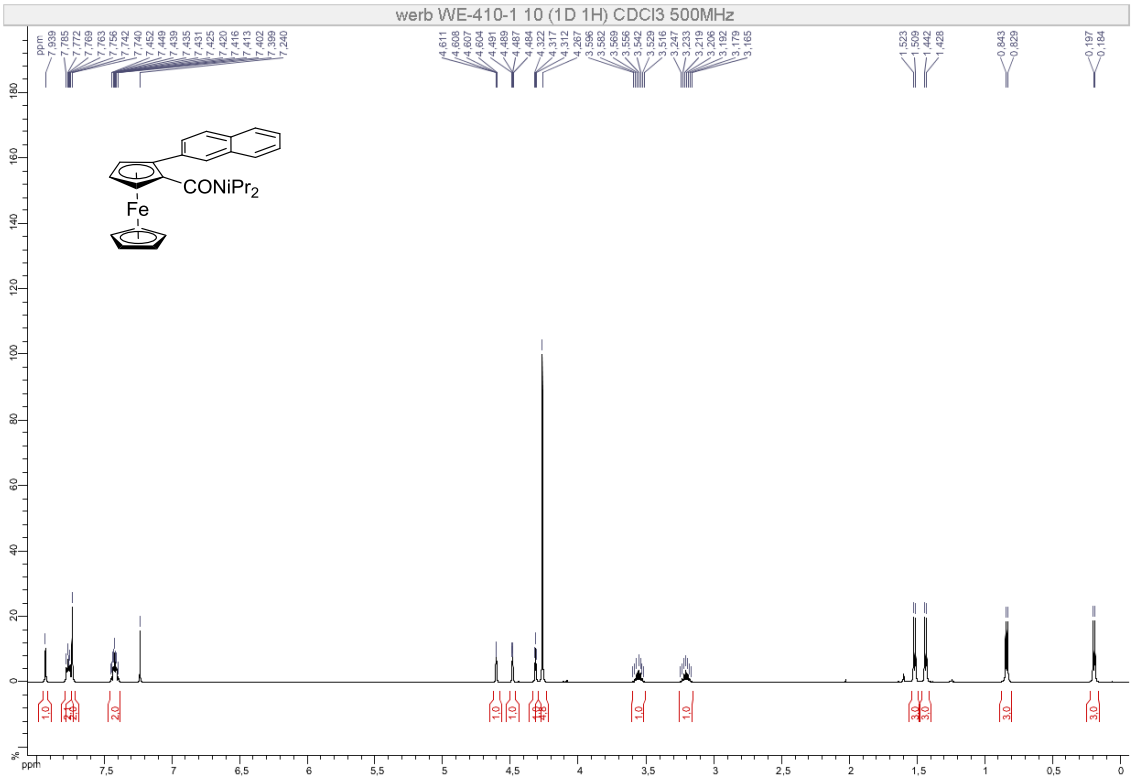

werb WE-399-1 13 (COSY 1H/1H) CDCl3 500MHz
MeO
OMe
CONiPr2
Fe
ppm

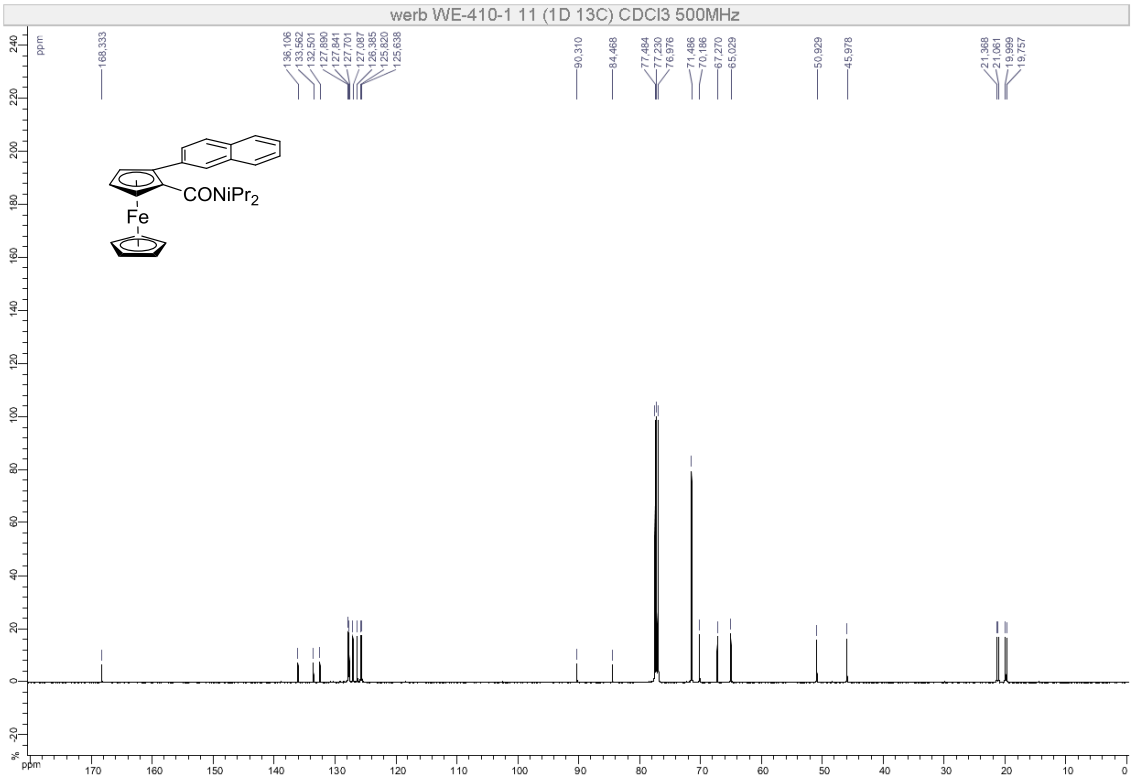

werb WE-399-1 14 (HSQC 1H/13C) CDCl3 500MHz
MeO
OMe
CONiPr2
Fe
ppm

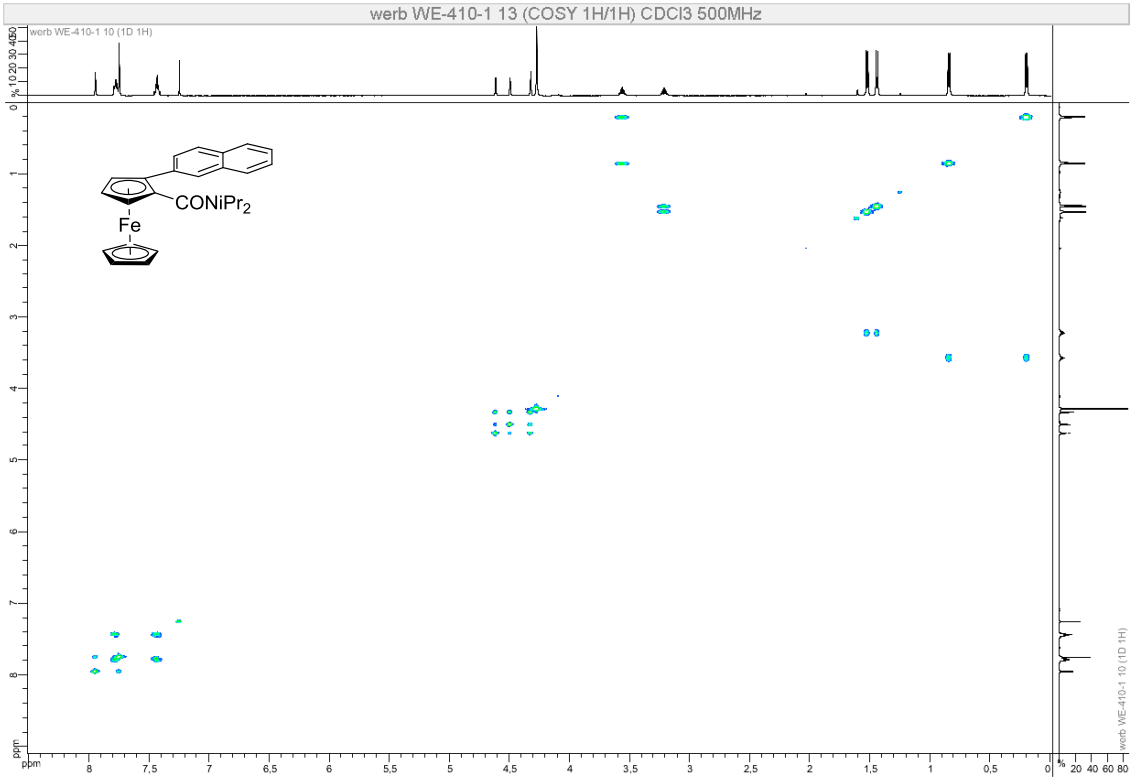

werb WE-399-1 15 (HMBC 1H/13C) CDCl3 500MHz
werb WE-399-1 10 (1D 1H)
MeO
OMe
CONiPr2
Fe
ppm
werb WE-399-1 11 (1D 13C)

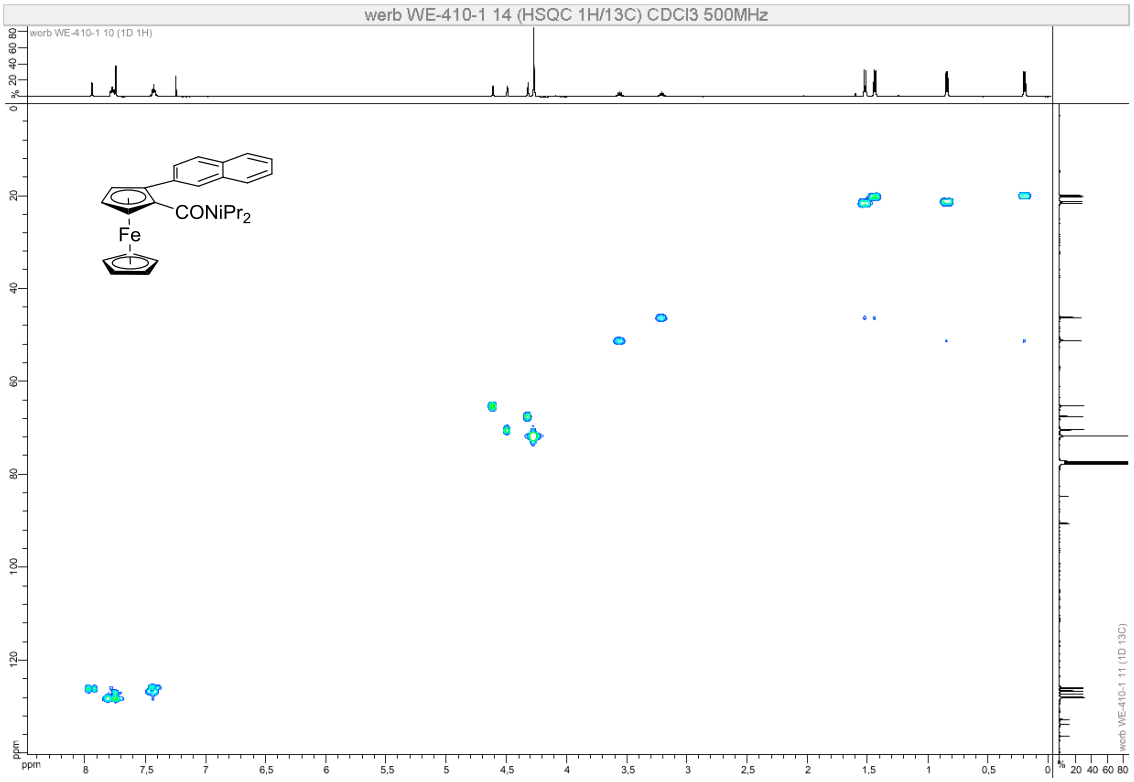

werb WE-399-1 16 (NOESY 1H/1H) CDCl3 500MHz
werb WE-399-1 10 (1D 1H)
MeO
OMe
CONiPr2
Fe
ppm
werb WE-399-1 10 (1D 1H)

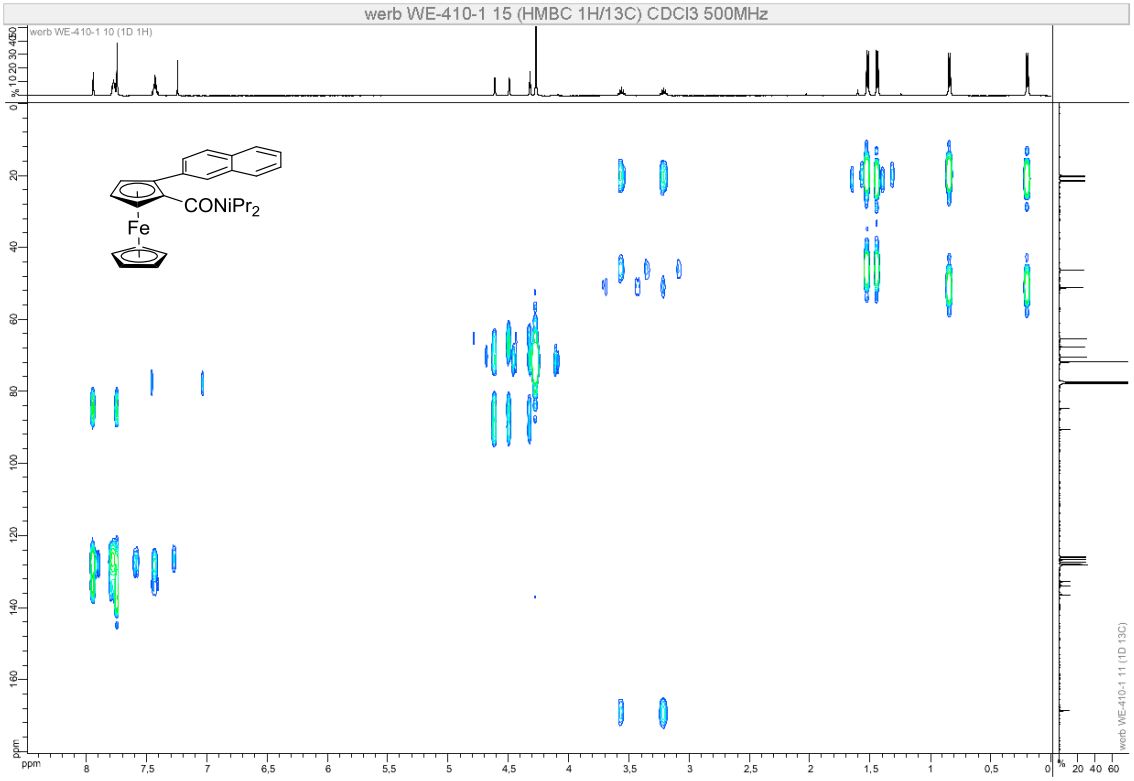
Compound (±)-7

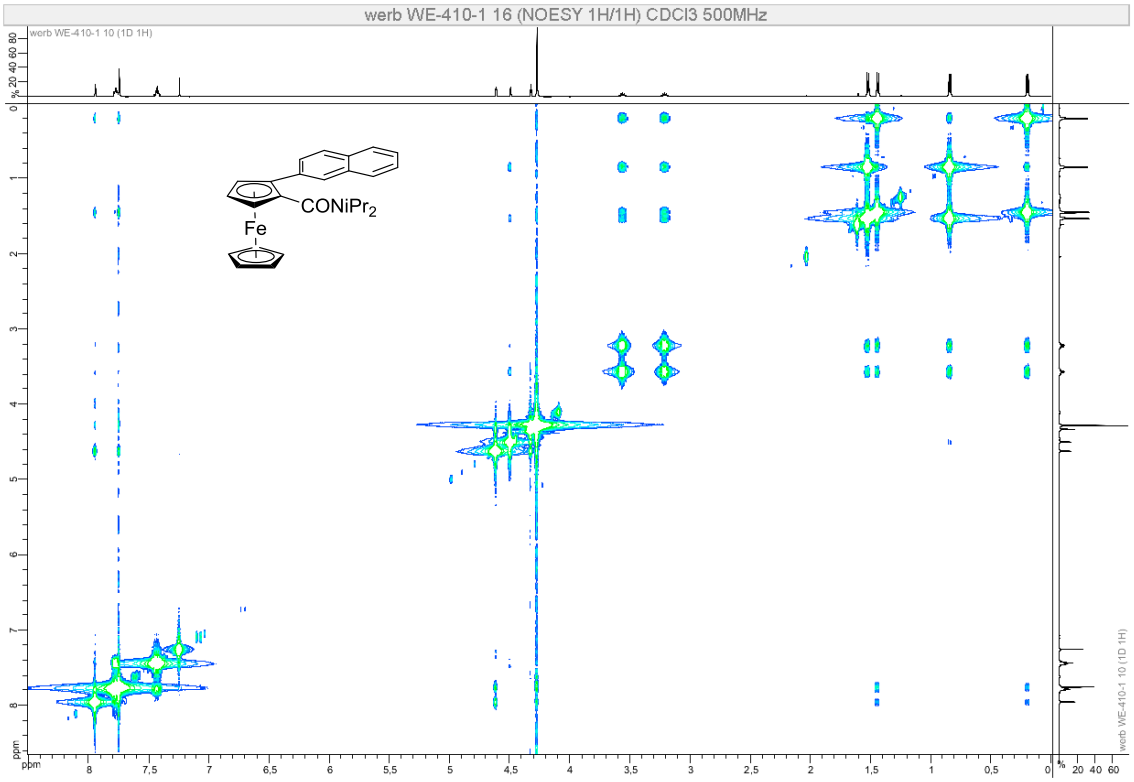

werb WE-410-1 10 (1D 1H) CDCl3 500MHz
CONiPr2
Fe


werb WE-410-1 11 (1D 13C) CDCl3 500MHz
CONiPr2
Fe

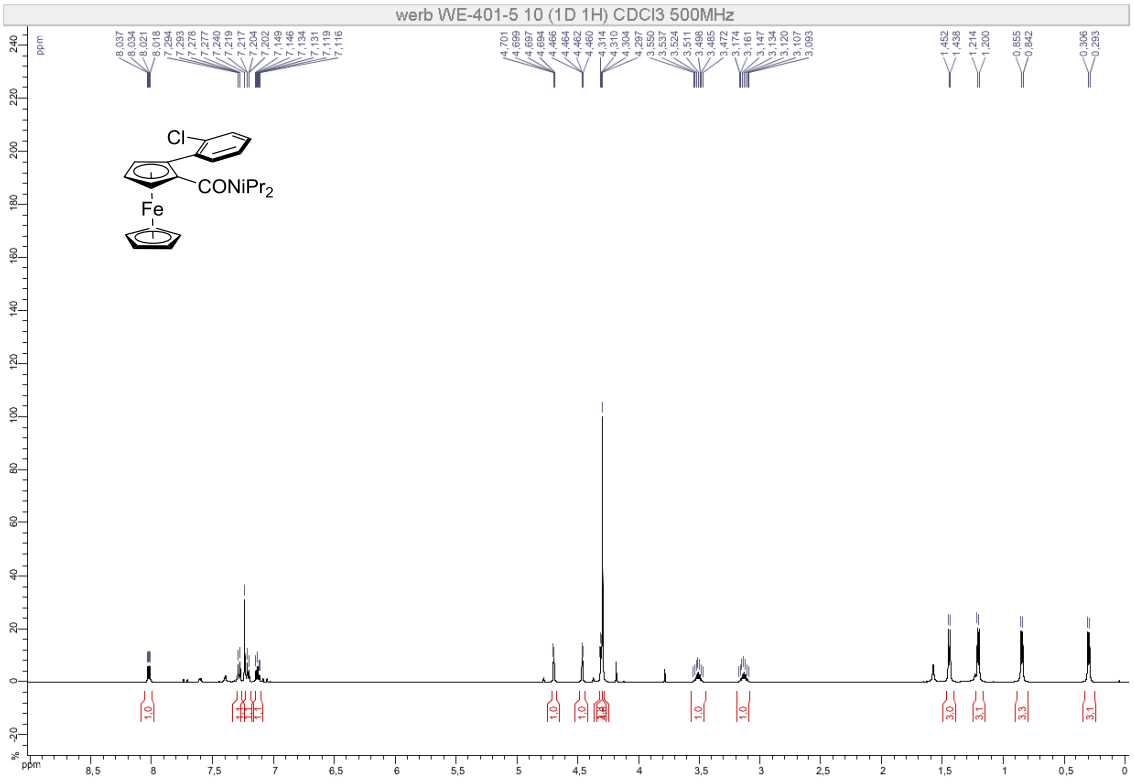

werb WE-410-1 13 (COSY 1H/1H) CDCl3 500MHz
CONiPr2
Fe
ppm

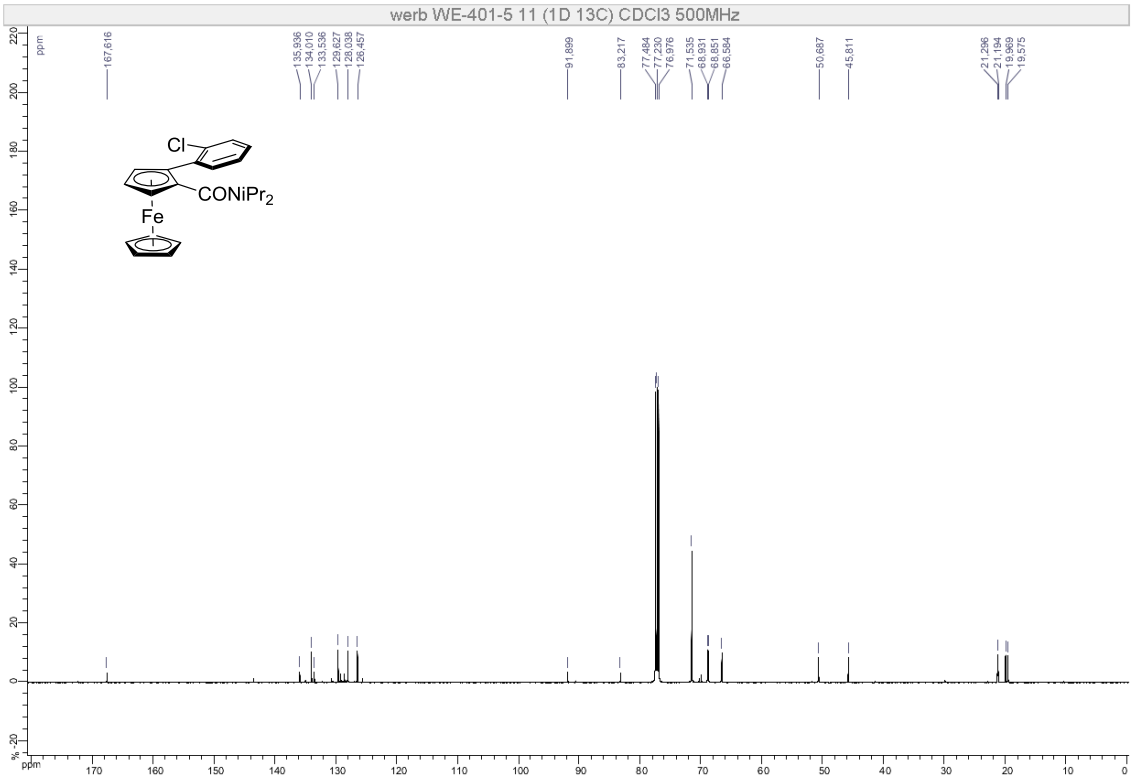

werb WE-410-1 14 (HSQC 1H/13C) CDCl3 500MHz
CONiPr2
Fe
ppm

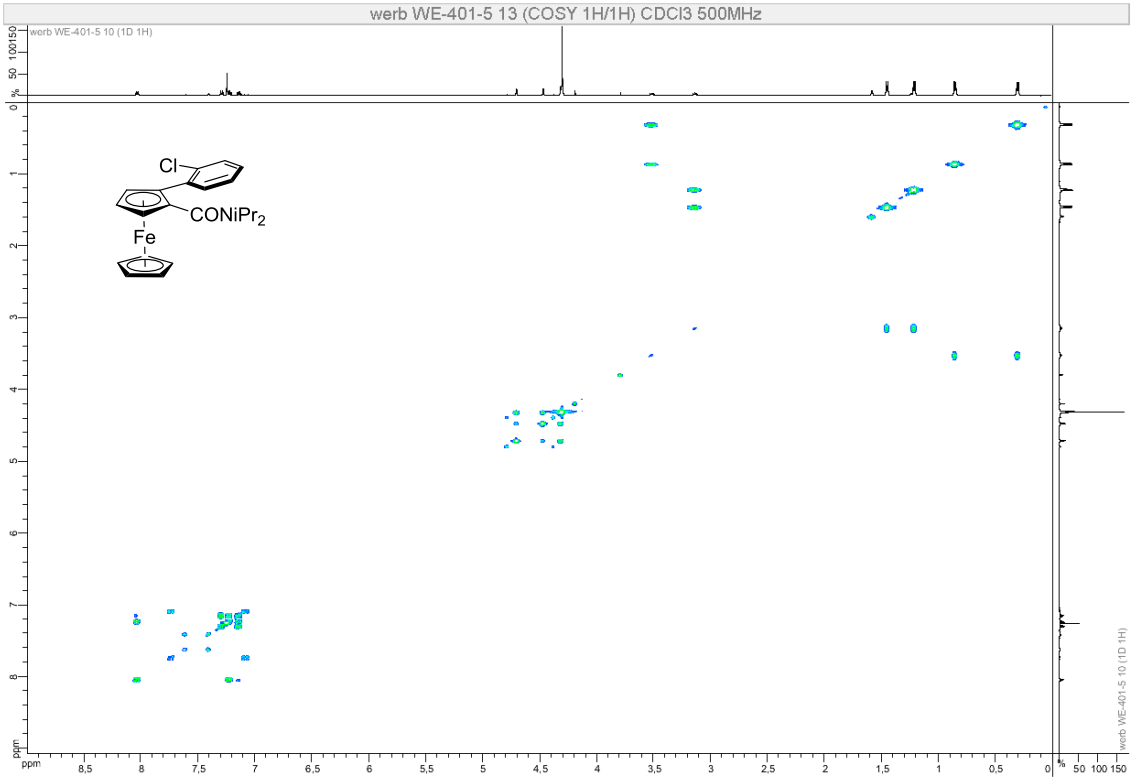

werb WE-410-1 15 (HMBC 1H/13C) CDCl3 500MHz
werb WE-410-1 10 (1D 1H)
CONiPr2
Fe
ppm
werb WE-410-1 11 (1D 13C)

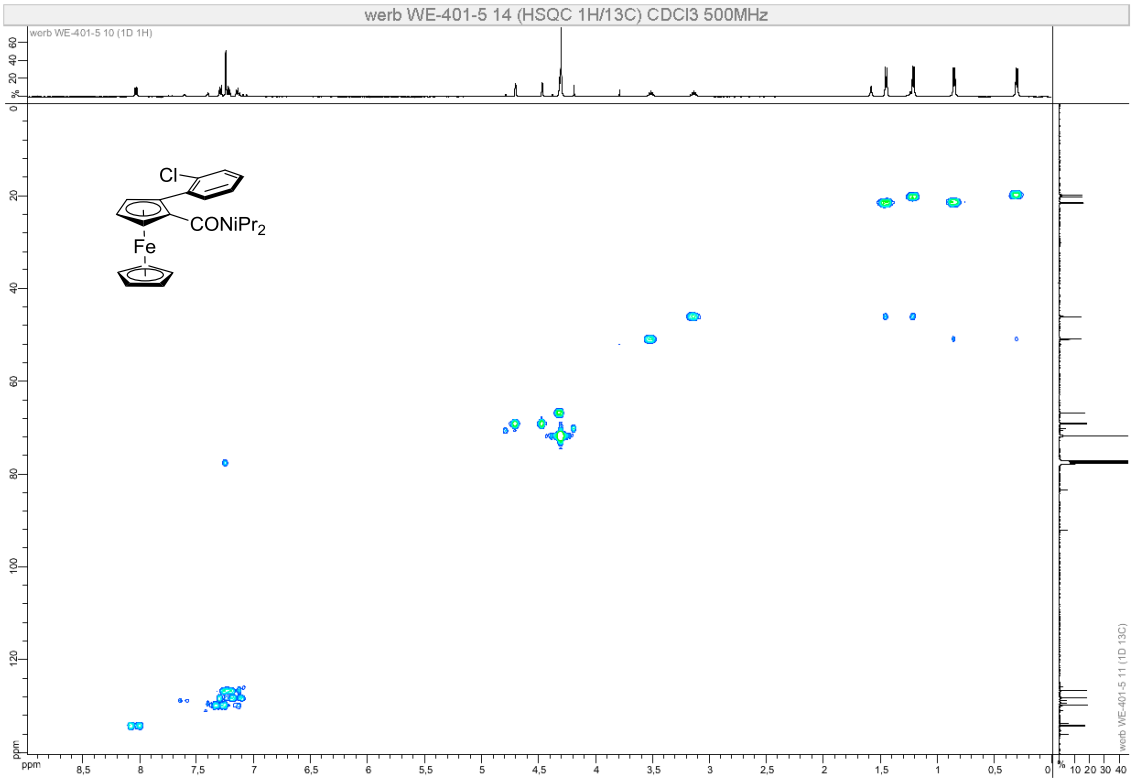

werb WE-410-1 16 (NOESY 1H/1H) CDCl3 500MHz
werb WE-410-1 10 (1D 1H)
CONiPr2
Fe
ppm
werb WE-410-1 10 (1D 1H)

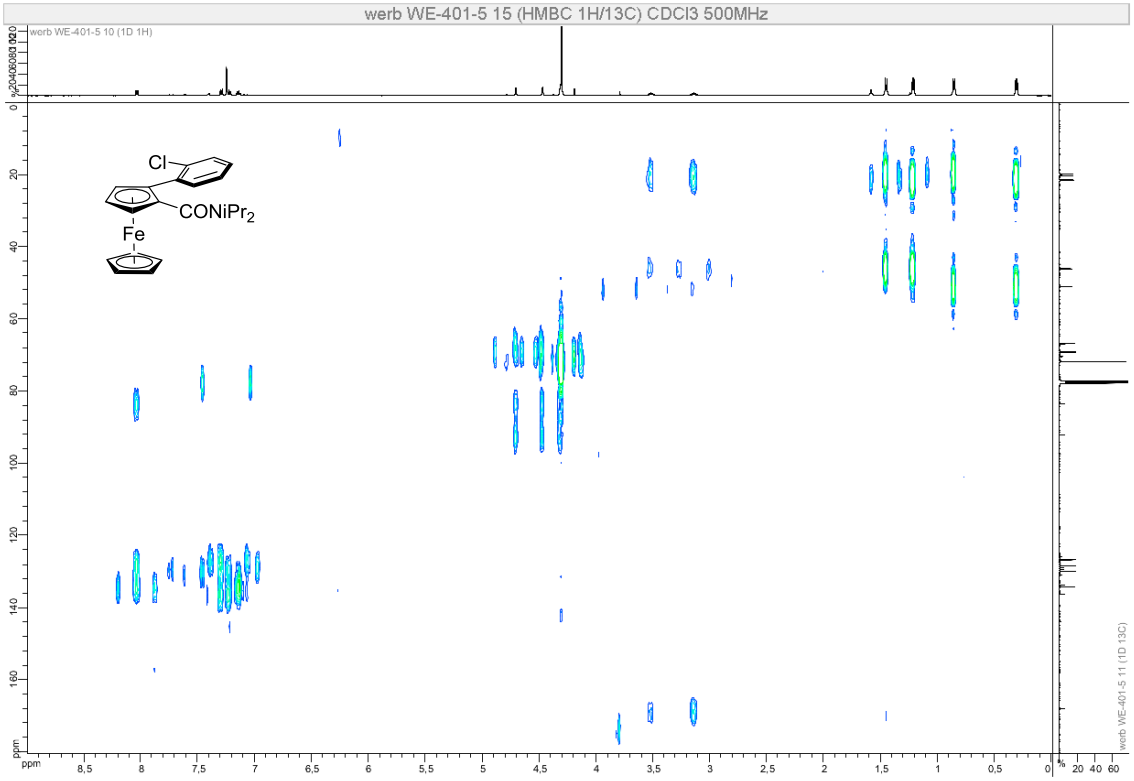
Compound (±)-8

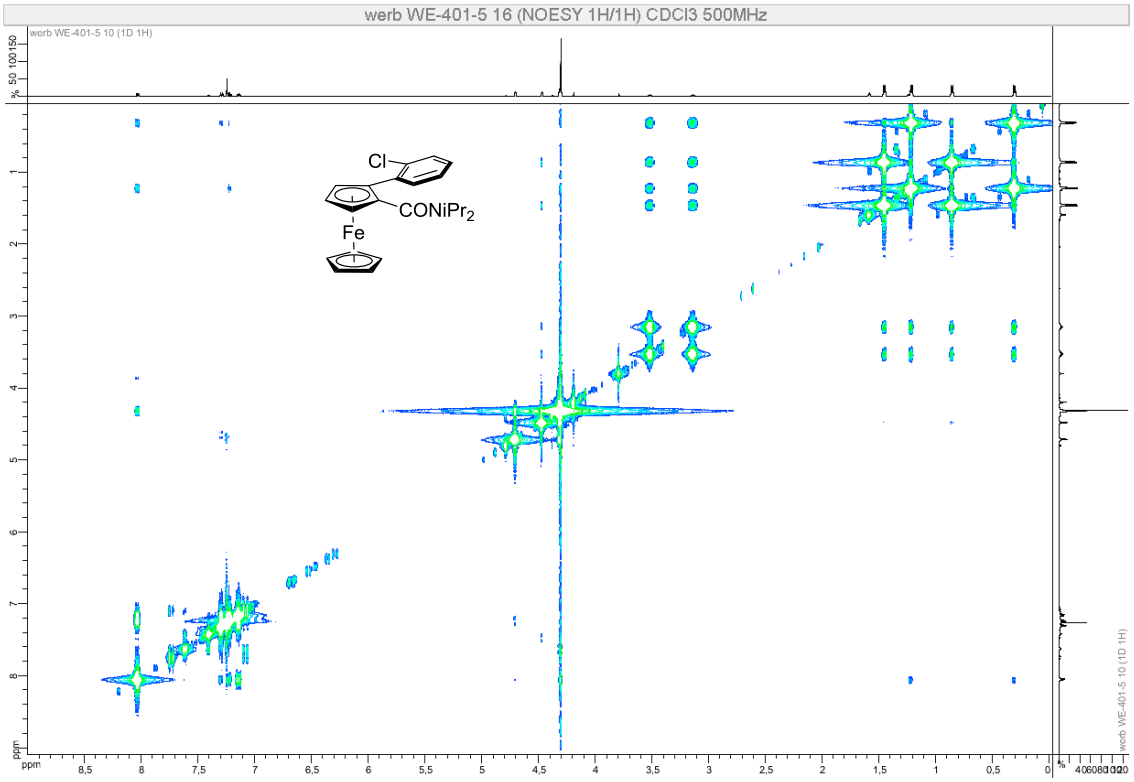

werb WE-401-5 10 (1D 1H) CDCl3 500MHz
Cl
CONiPr2
Fe


werb WE-401-5 11 (1D 13C) CDCl3 500MHz
Cl
CONiPr2
Fe

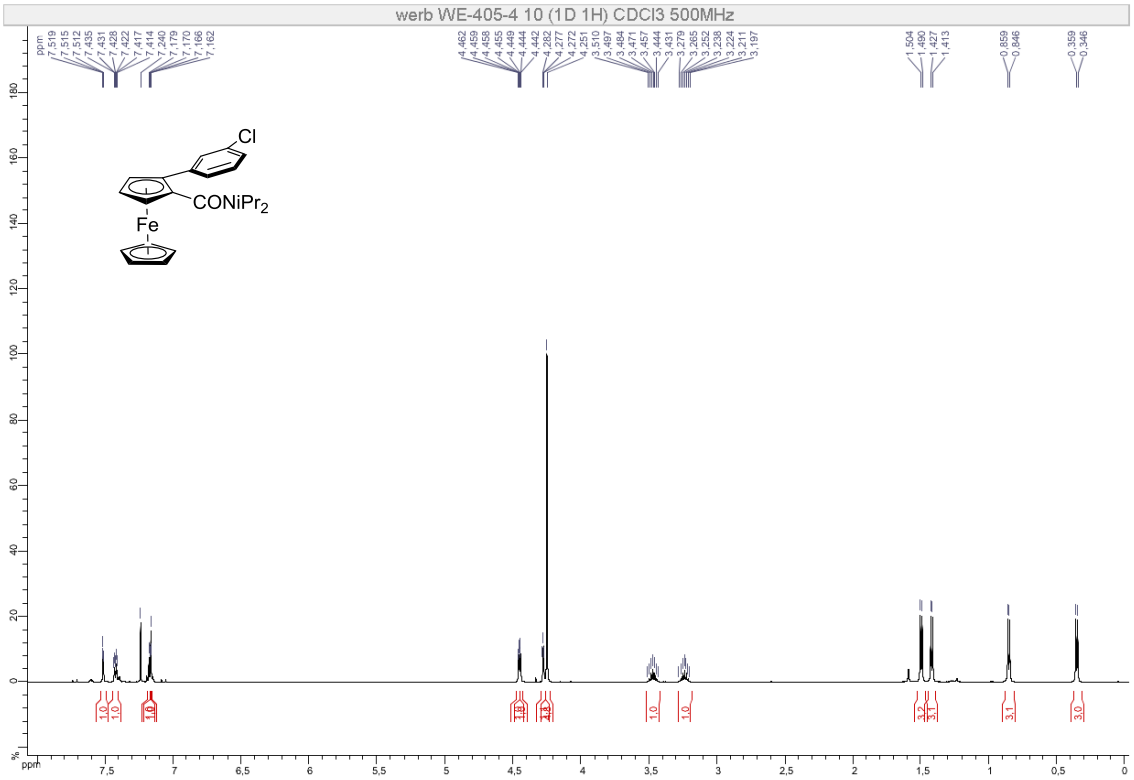

werb WE-401-5 13 (COSY 1H/1H) CDCl3 500MHz
werb WE-401-5 10 (1D 1H)
Cl
CONiPr2
Fe
ppm
werb WE-401-5 10 (1D 1H)

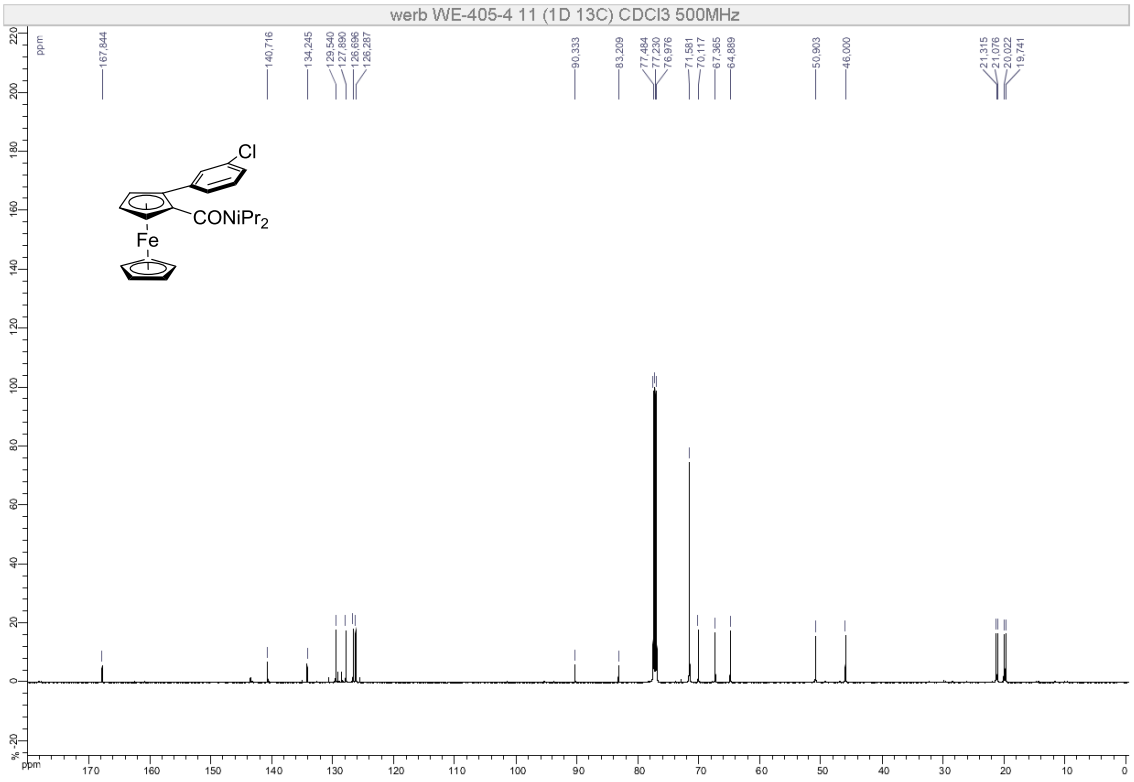

werb WE-401-5 14 (HSQC 1H/13C) CDCl3 500MHz
werb WE-401-5 10 (1D 1H)
Cl
CONiPr2
Fe
ppm
werb WE-401-5 11 (1D 13C)

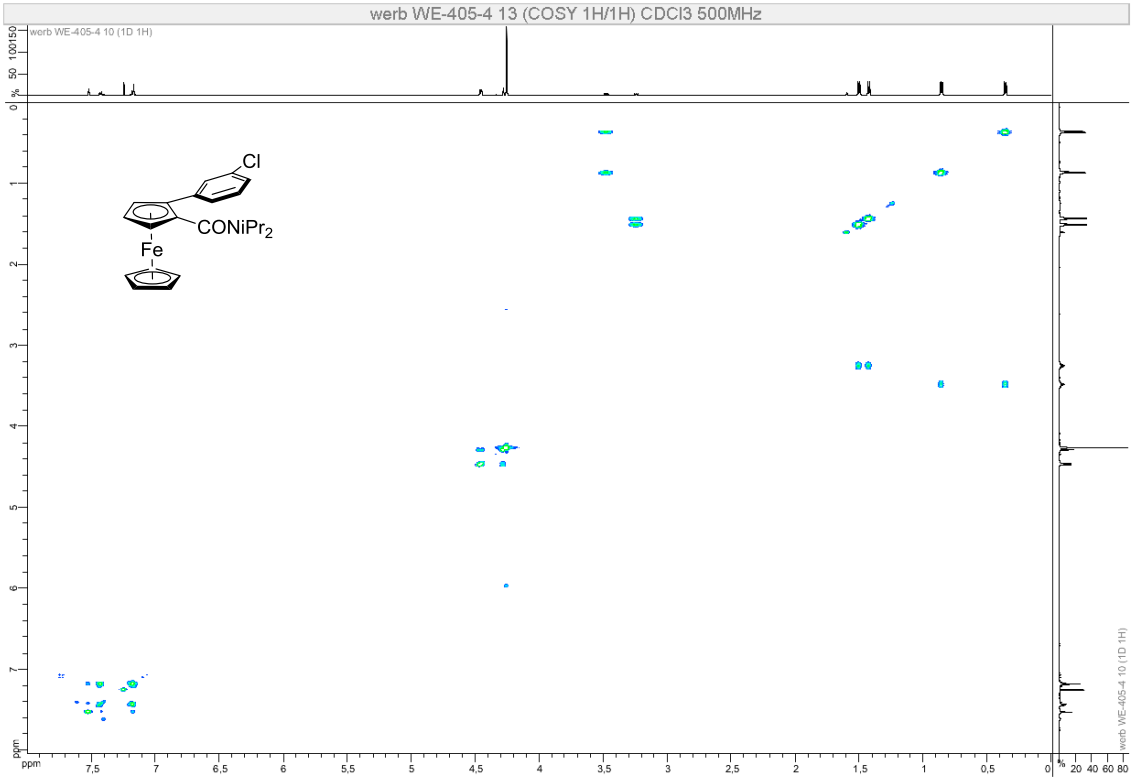

werb WE-401-5 15 (HMBC 1H/13C) CDCl3 500MHz
werb WE-401-5 10 (1D 1H)
Cl
CONiPr2
Fe
ppm
werb WE-401-5 11 (1D 13C)

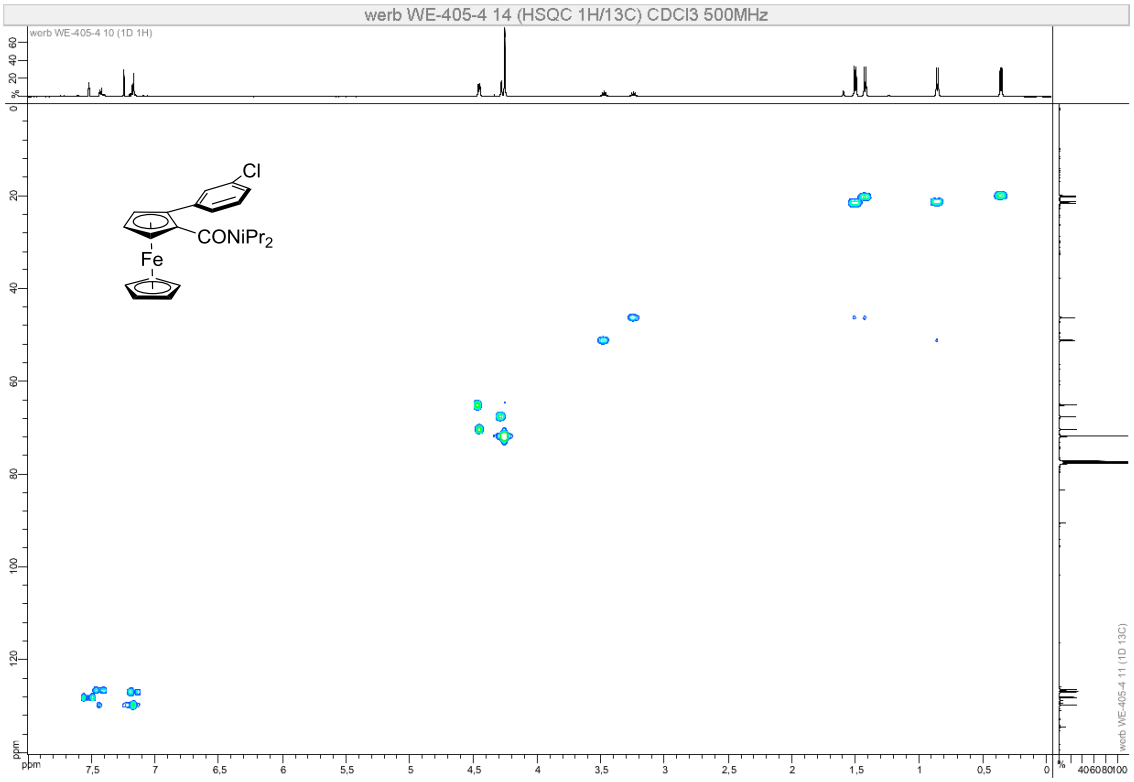

werb WE-401-5 16 (NOESY 1H/1H) CDCl3 500MHz
werb WE-401-5 10 (1D 1H)
Cl
CONiPr2
Fe
ppm
werb WE-401-5 10 (1D 1H)

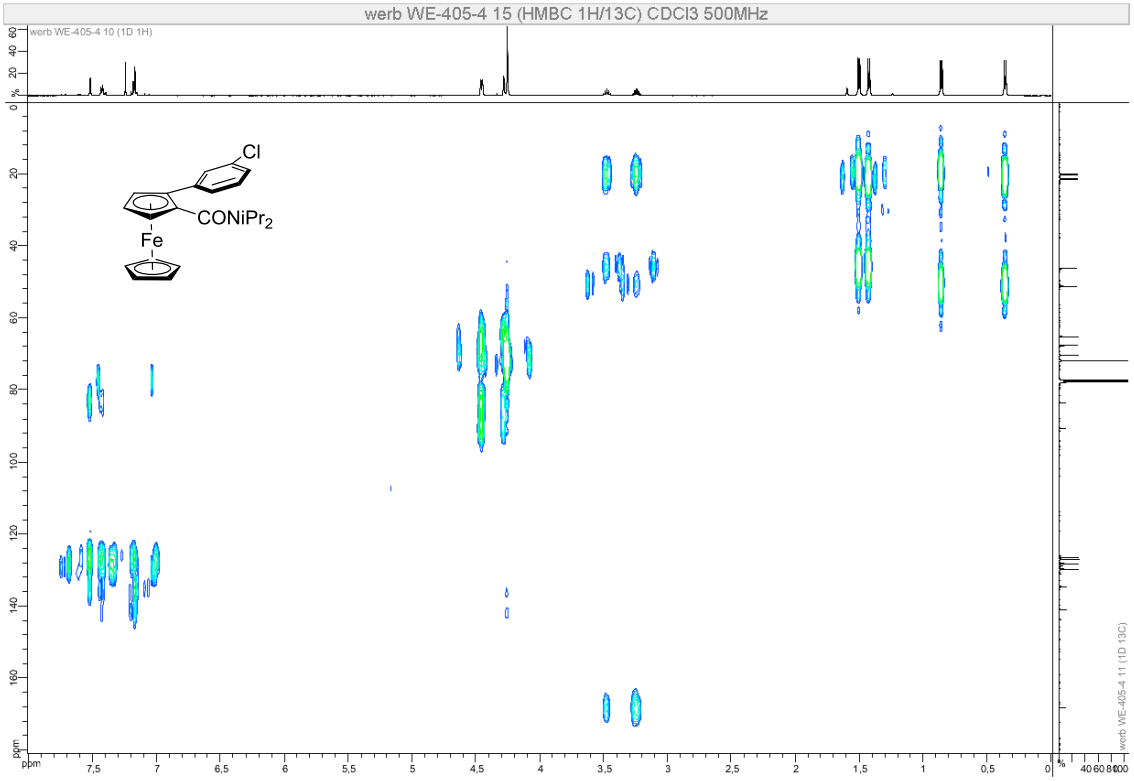
Compound (±)-9

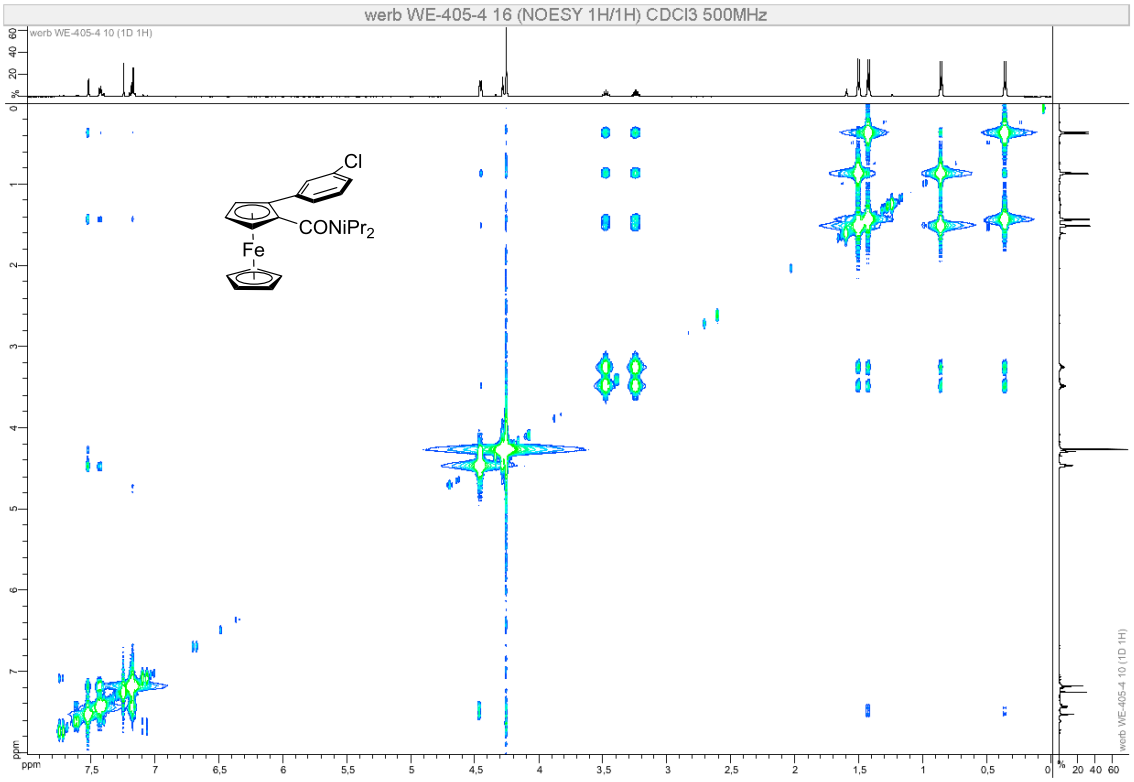

werb WE-405-4 10 (1D 1H) CDCl3 500MHz
Cl
CONiPr2
Fe


werb WE-405-4 11 (1D 13C) CDCl3 500MHz
Cl
CONiPr2
Fe

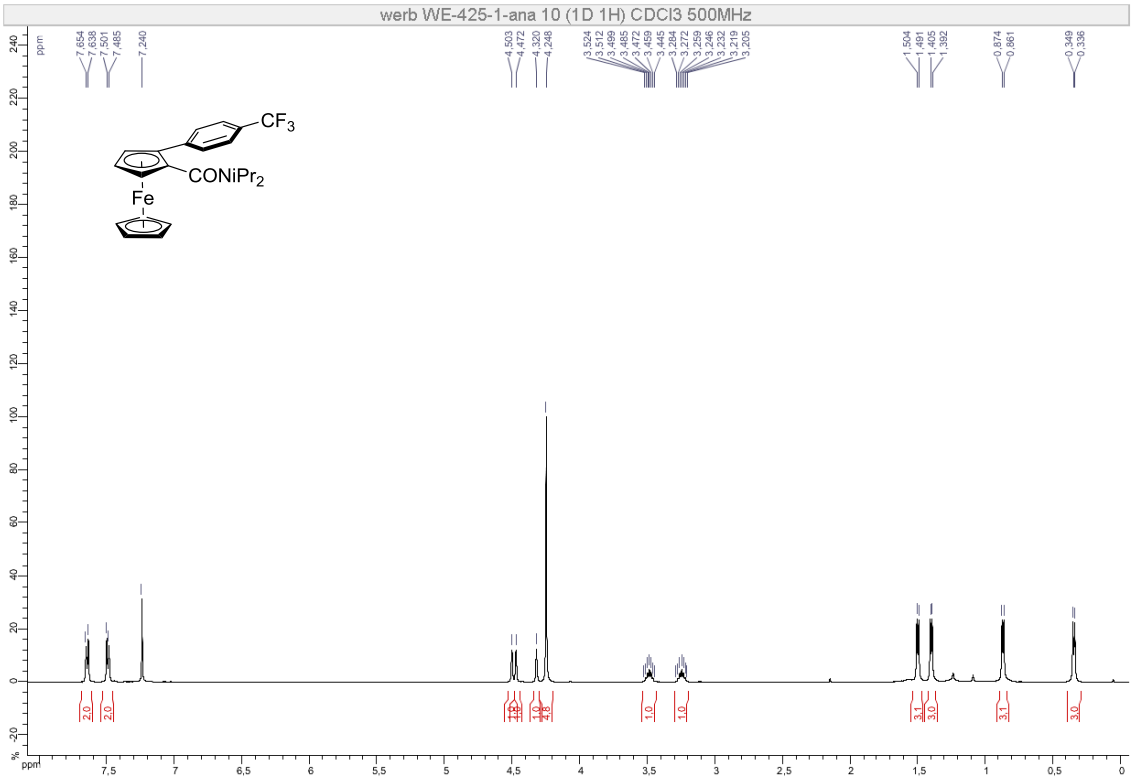

werb WE-405-4 13 (COSY 1H/1H) CDCl3 500MHz
Cl
CONiPr2
Fe
ppm

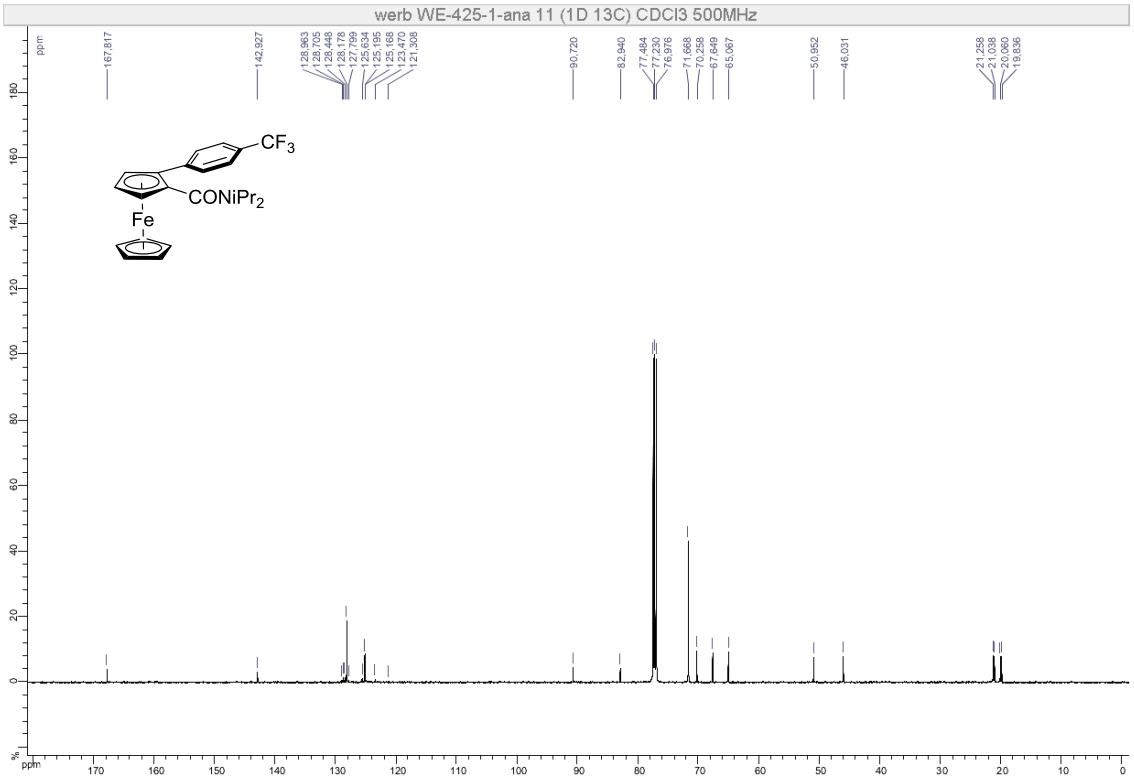

werb WE-405-4 14 (HSQC 1H/13C) CDCl3 500MHz
Cl
CONiPr2
Fe
ppm

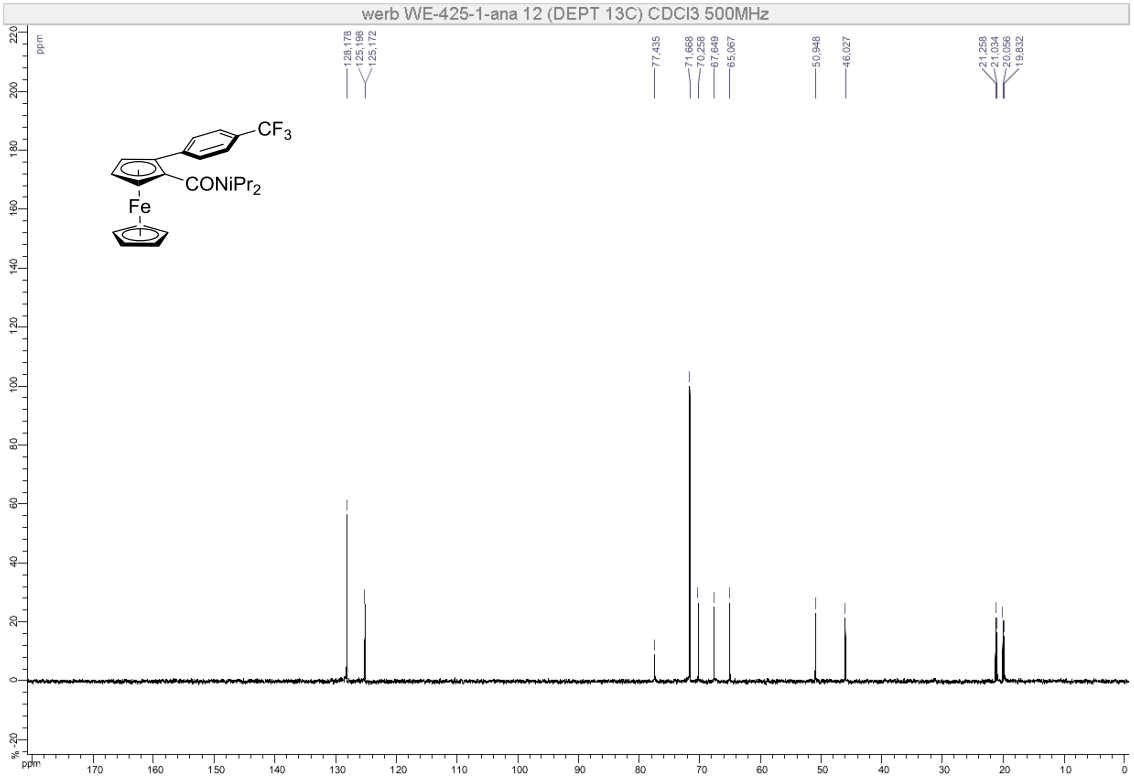

werb WE-405-4 15 (HMBC 1H/13C) CDCl3 500MHz
werb WE-405-4 10 (1D 1H)
Cl
CONiPr2
Fe
ppm
werb WE-405-4 11 (1D 13C)

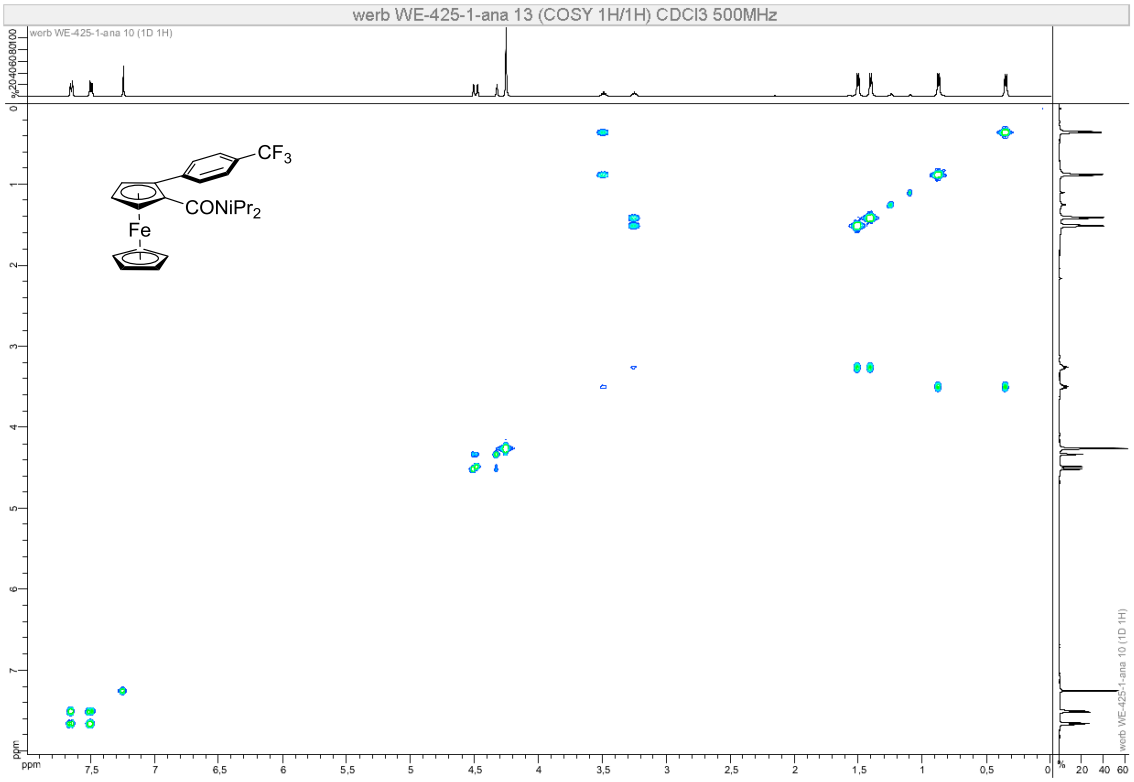

werb WE-405-4 16 (NOESY 1H/1H) CDCl3 500MHz
werb WE-405-4 10 (1D 1H)
Cl
CONiPr2
Fe
ppm
werb WE-405-4 10 (1D 1H)

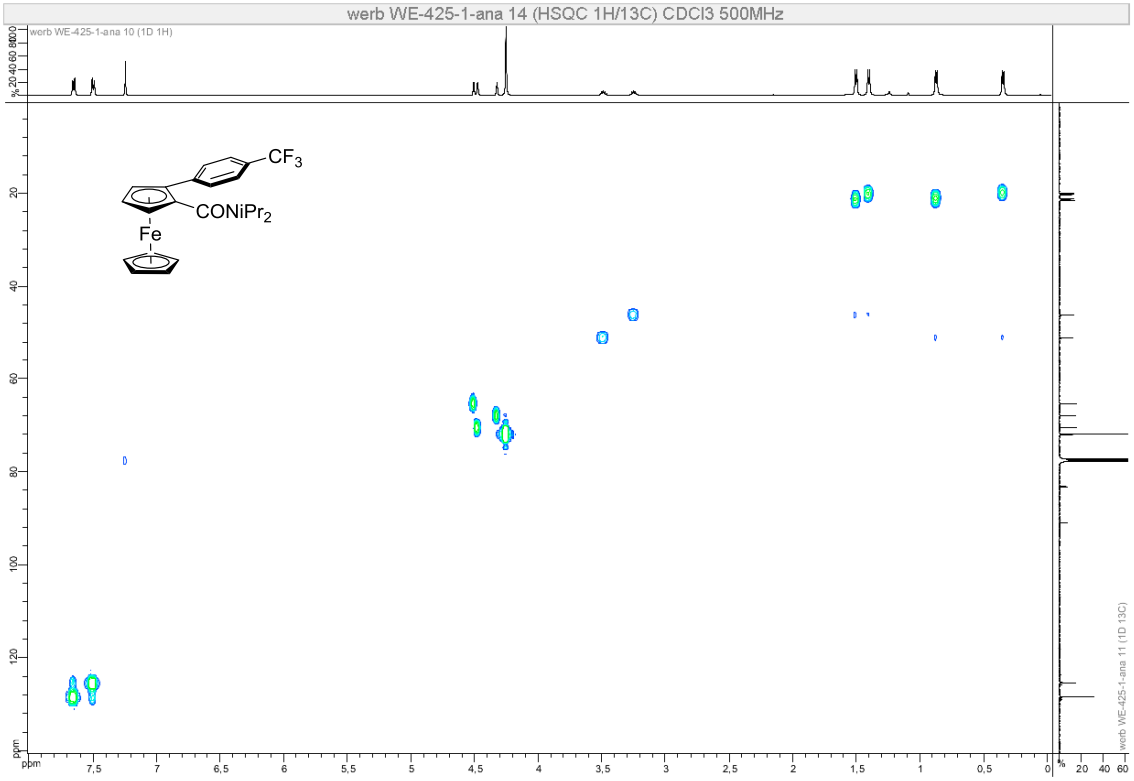
Compound (±)-10

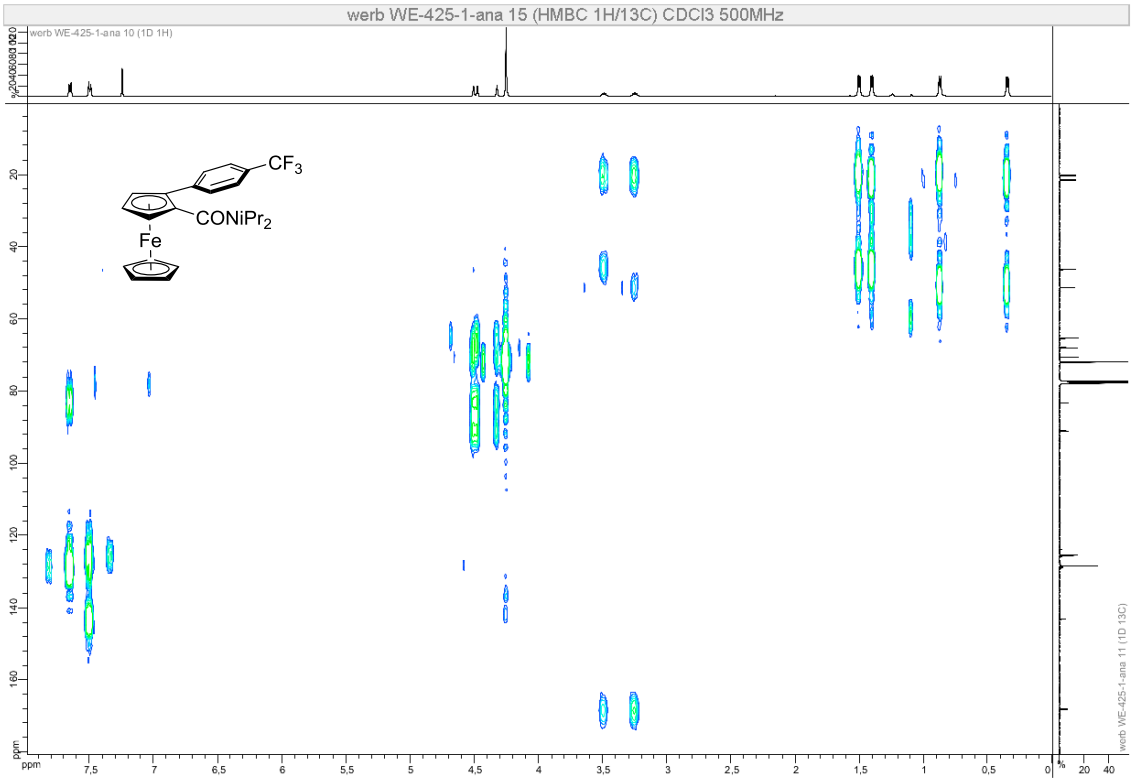

werb WE-425-1-ana 10 (1D 1H) CDCl3 500MHz
CF3
CONiPr2
Fe

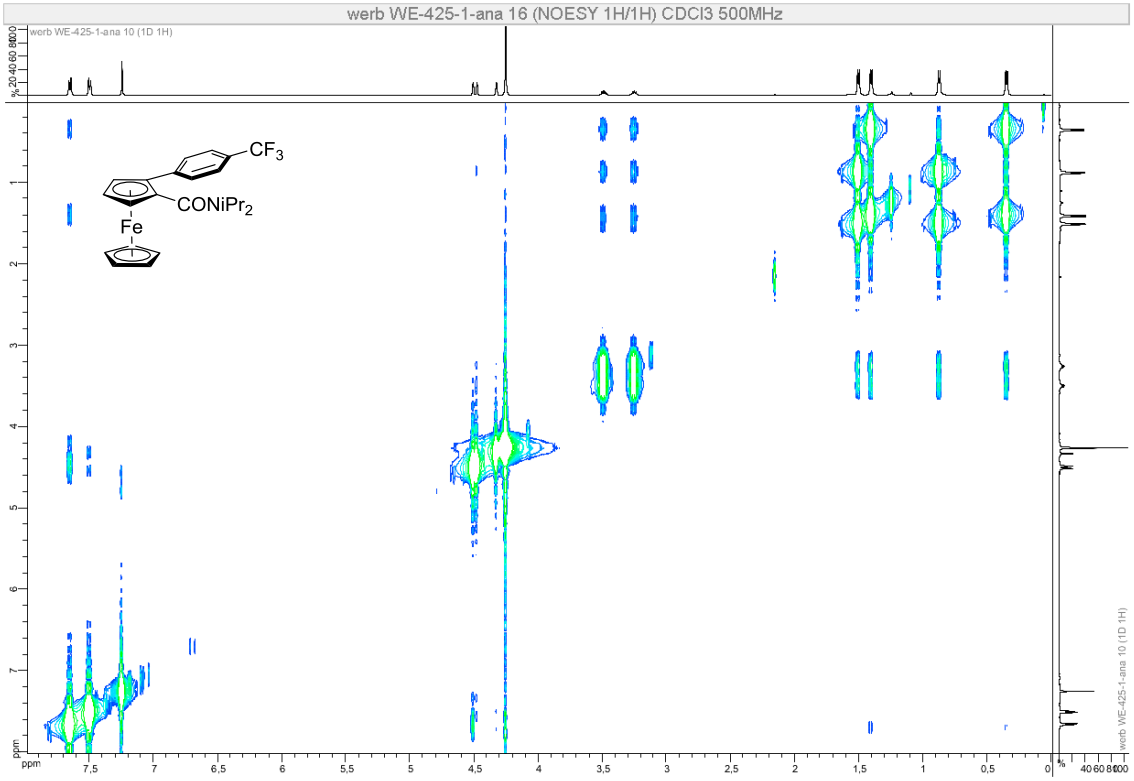

werb WE-425-1-ana 11 (1D 13C) CDCl3 500MHz
CF3
CONiPr2
Fe

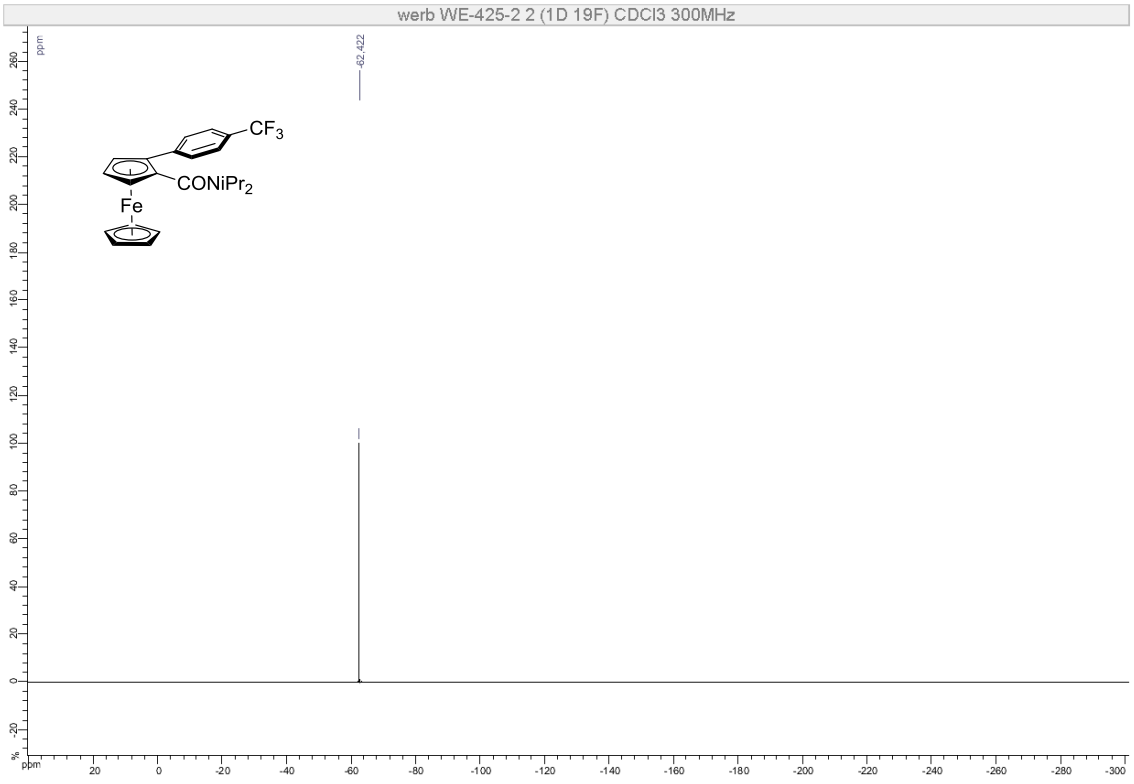

werb WE-425-1-ana 12 (DEPT 13C) CDCl3 500MHz
ppm
128,178
125,198
125,172
77,435
71,668
70,258
67,649
65,067
50,948
46,027
21,258
21,034
20,955
19,832
CF3
CONiPr2
Fe


werb WE-425-1-ana 13 (COSY 1H/1H) CDCl3 500MHz
werb WE-425-1-ana 10 (1D 1H)
CF3
CONiPr2
Fe
ppm
werb WE-425-1-ana 10 (1D 1H)

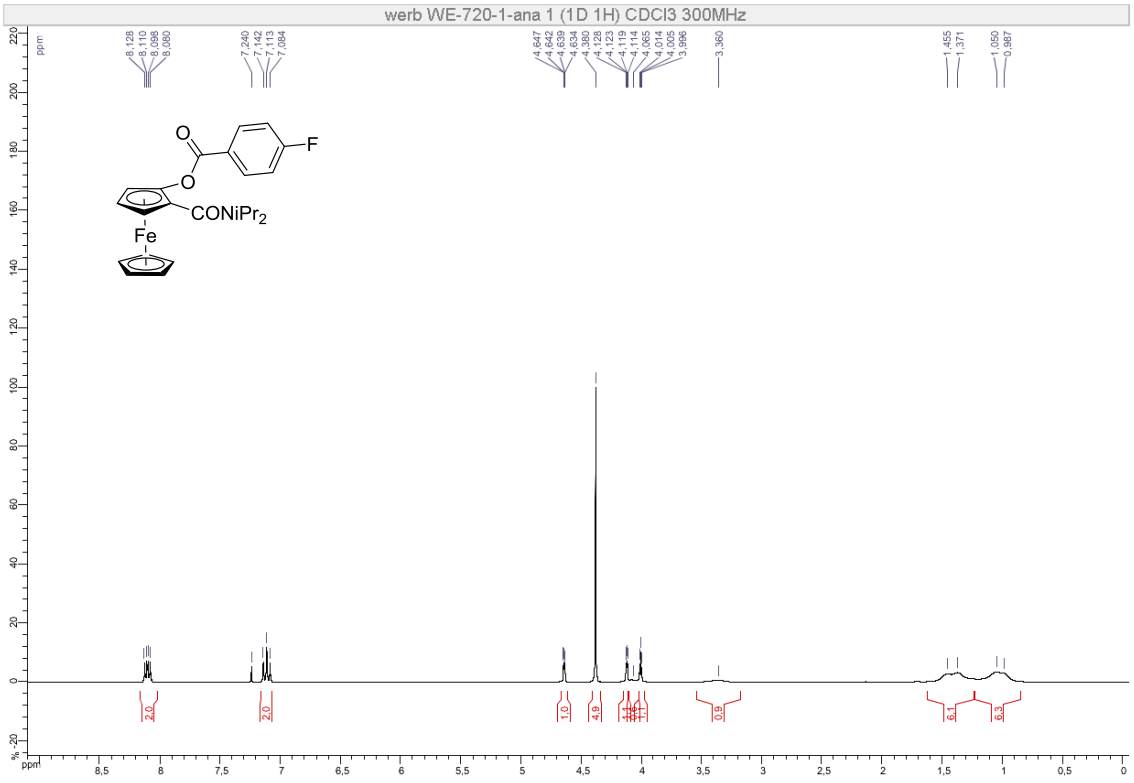

werb WE-425-1-ana 14 (HSQC 1H/13C) CDCl3 500MHz
werb WE-425-1-ana 10 (1D 1H)
CF3
CONiPr2
Fe
ppm
werb WE-425-1-ana 11 (1D 13C)

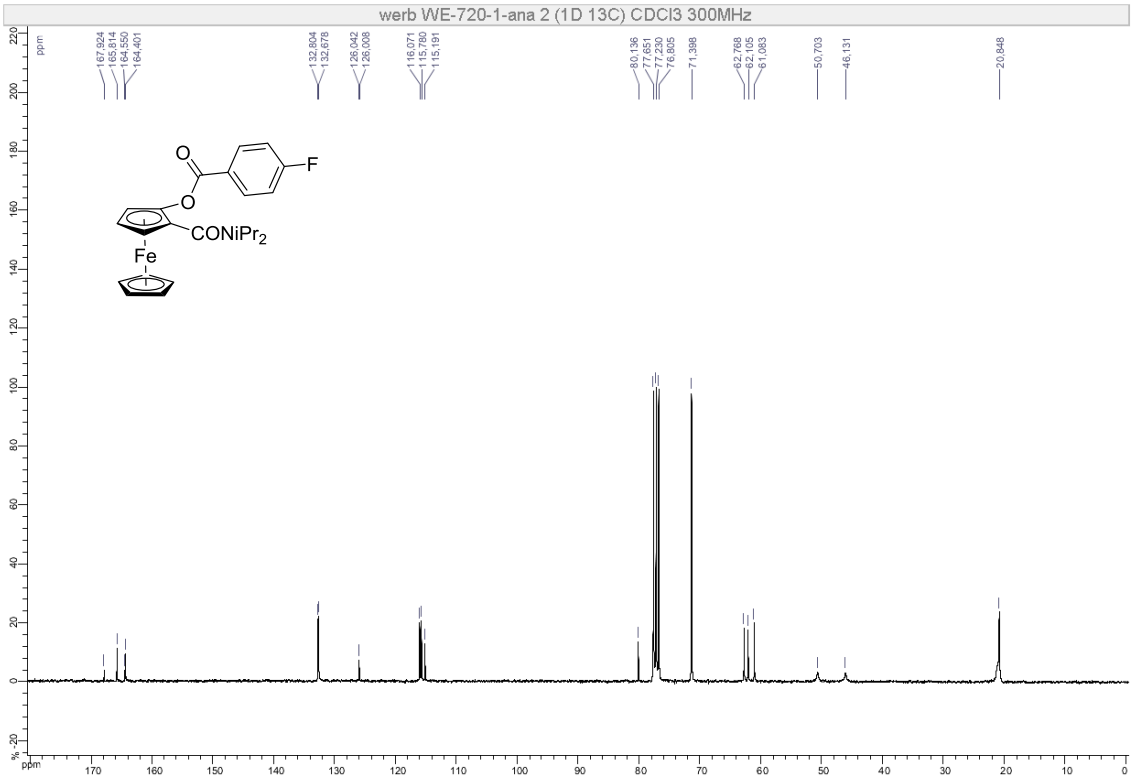

werb WE-425-1-ana 15 (HMBC 1H/13C) CDCl3 500MHz
werb WE-425-1-ana 10 (1D 1H)
CF3
CONiPr2
Fe
ppm
werb WE-425-1-ana 11 (1D 13C)

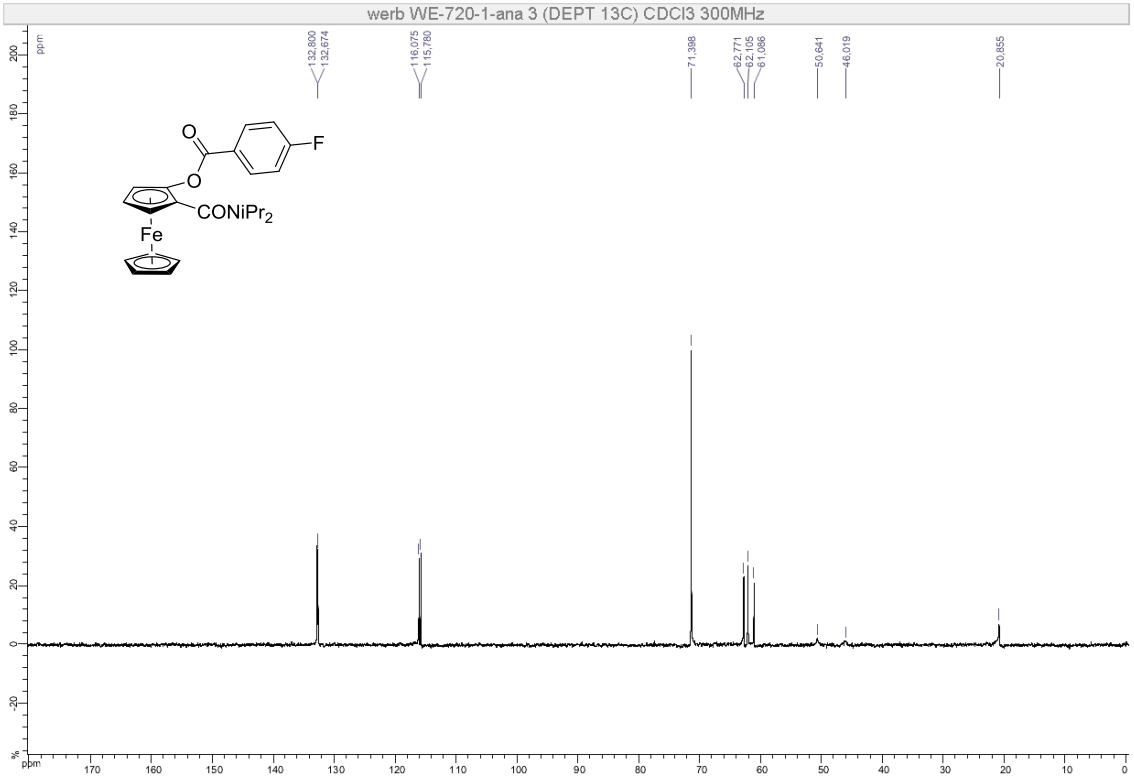

werb WE-425-1-ana 16 (NOESY 1H/1H) CDCl3 500MHz
werb WE-425-1-ana 10 (1D 1H)
CF3
CONiPr2
Fe
ppm
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5
0
werb WE-425-1-ana 10 (1D 1H)

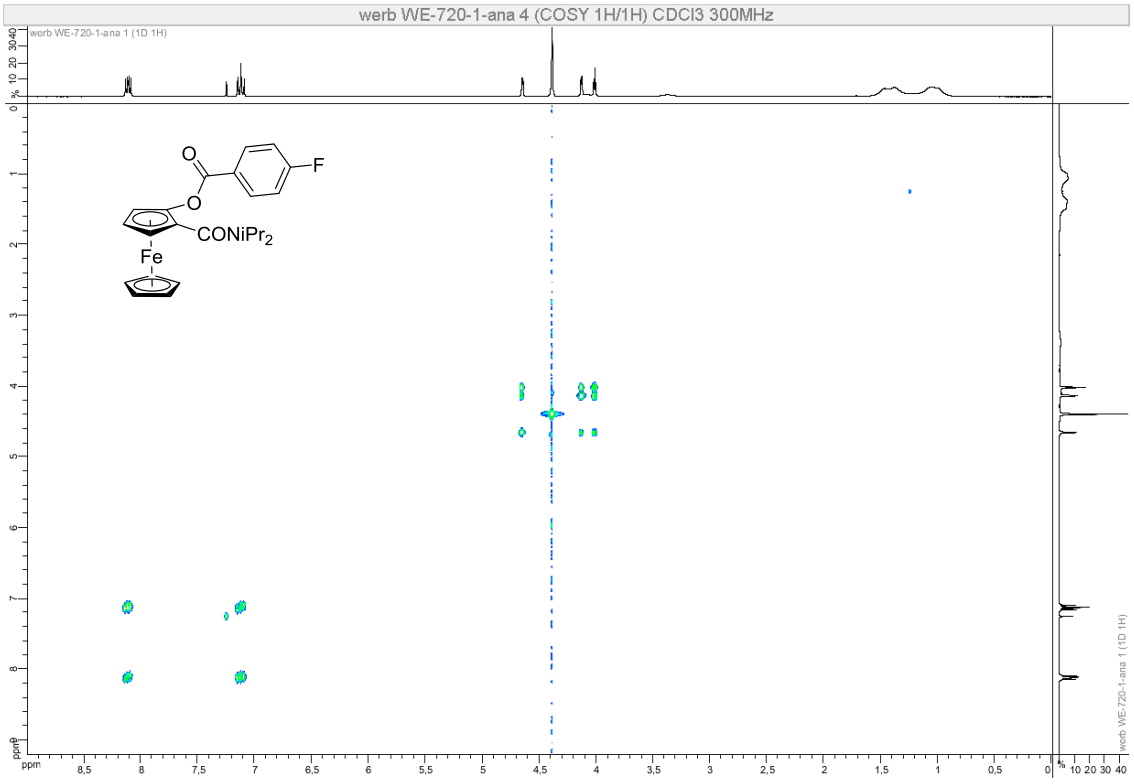

werb WE-425-2 2 (1D 19F) CDCl3 300MHz
ppm
-62.422
CF3
CONiPr2
Fe
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
-300

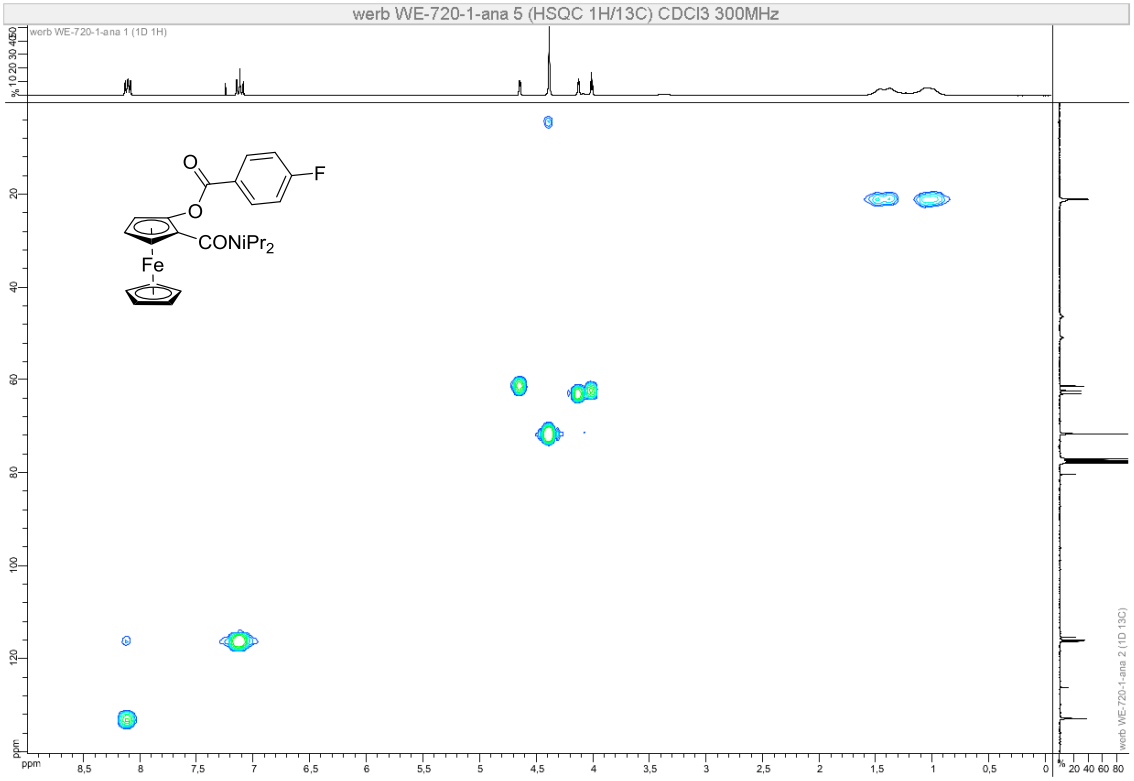
Compound (±)-11

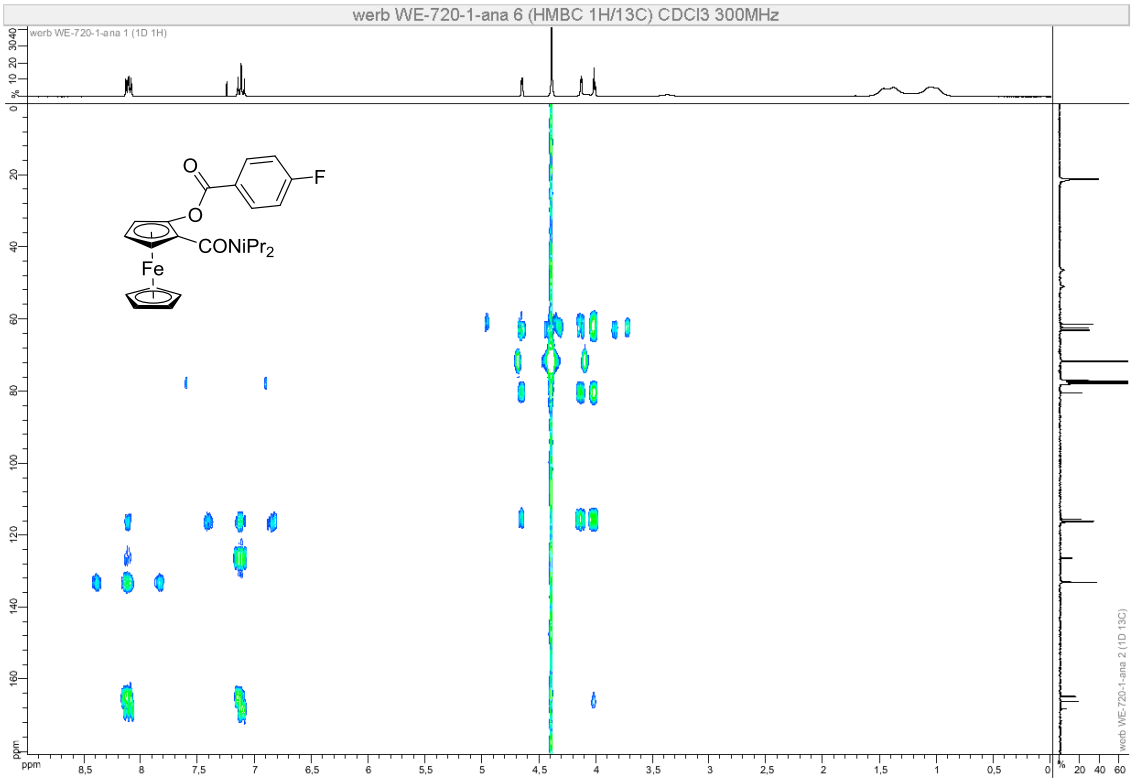

werb WE-720-1-ana 1 (1D 1H) CDCl3 300MHz
ppm
8.128
8.110
8.098
8.080
7.240
7.142
7.113
7.084
4.647
4.642
4.639
4.634
4.380
4.128
4.123
4.119
4.114
4.065
4.014
4.005
3.998
3.360
1.455
1.371
1.050
0.987
O
F
O
CONiPr2
Fe
2.0
2.0
1.0
4.9
0.9
6.1
6.3

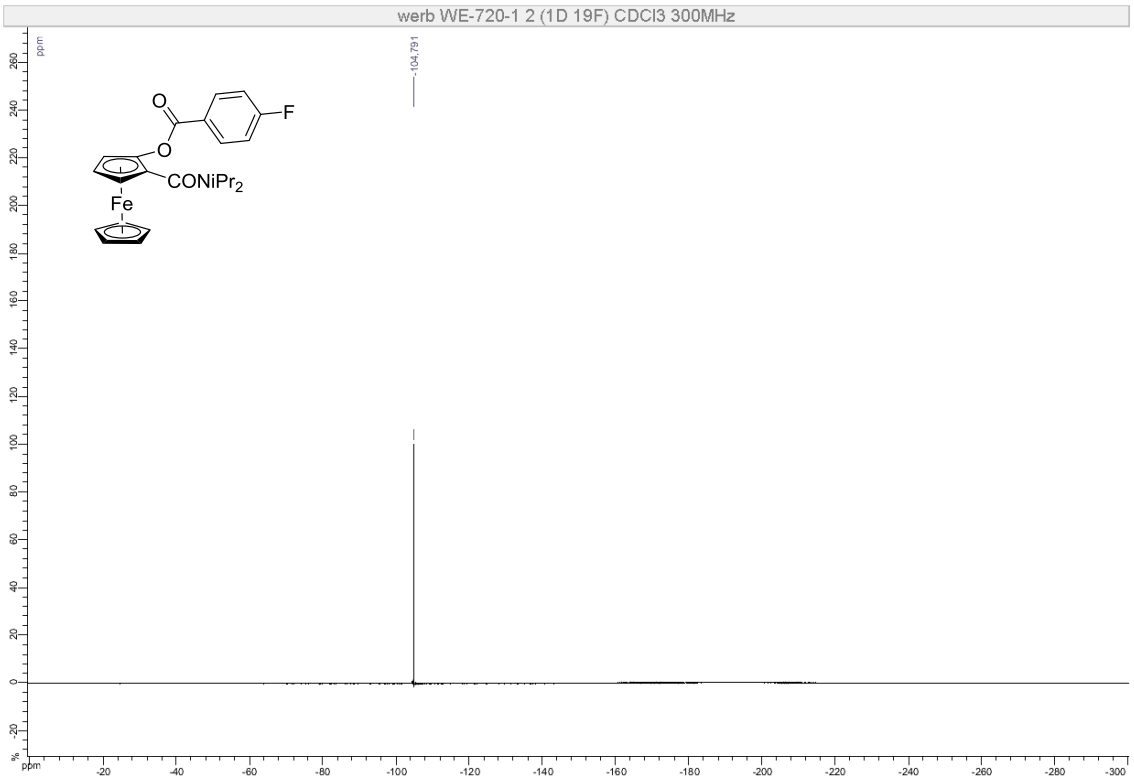

werb WE-720-1-ana 2 (1D 13C) CDCl3 300MHz
ppm
167.924
165.814
164.553
164.401
132.804
132.678
126.042
126.008
116.071
115.780
115.191
80.136
77.651
77.230
76.805
71.398
62.768
62.105
61.083
50.703
46.131
20.848
O
F
O
CONiPr2
Fe


werb WE-720-1-ana 3 (DEPT 13C) CDCl3 300MHz
132,800
132,674
116,075
115,780
71,398
62,771
62,105
61,086
50,641
46,019
20,855
O
F
O
CONiPr2
Fe

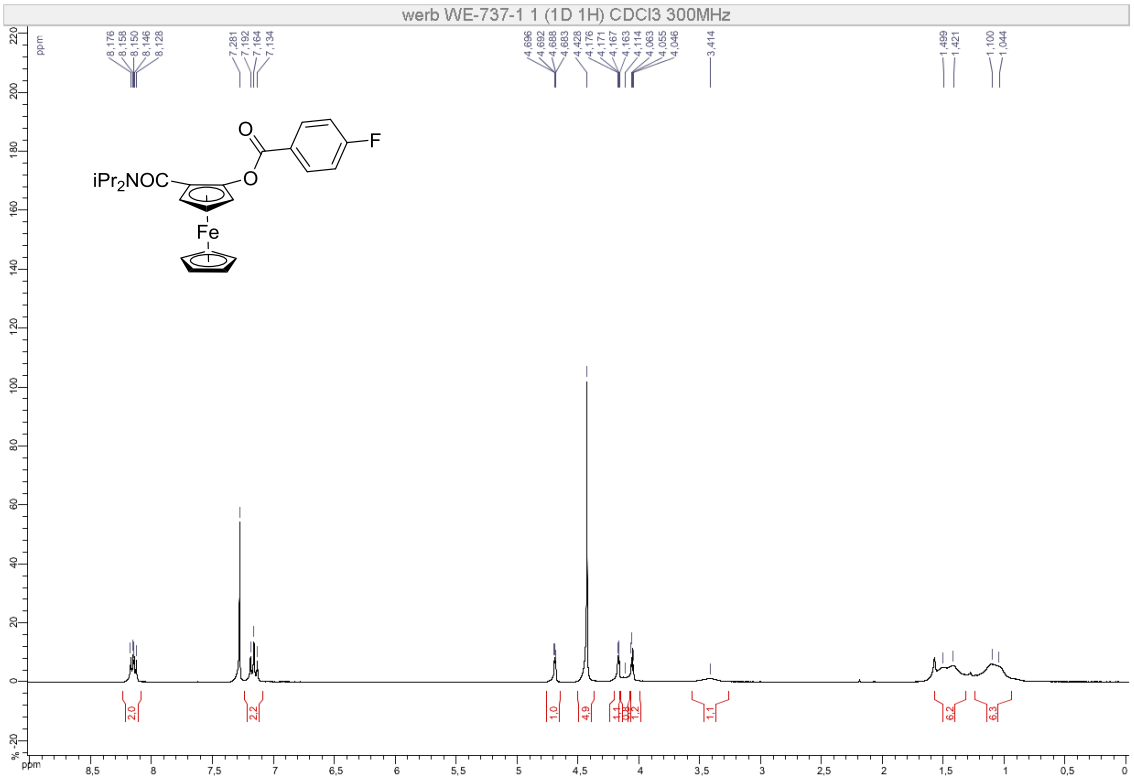

werb WE-720-1-ana 4 (COSY 1H/1H) CDCl3 300MHz
werb WE-720-1-ana 1 (1D 1H)
O
F
O
CONiPr2
Fe


werb WE-720-1-ana 5 (HSQC 1H/13C) CDCl3 300MHz
werb WE-720-1-ana 1 (1D 1H)
O
F
O
CONiPr2
Fe
ppm
werb WE-720-1-ana 2 (1D 13C)

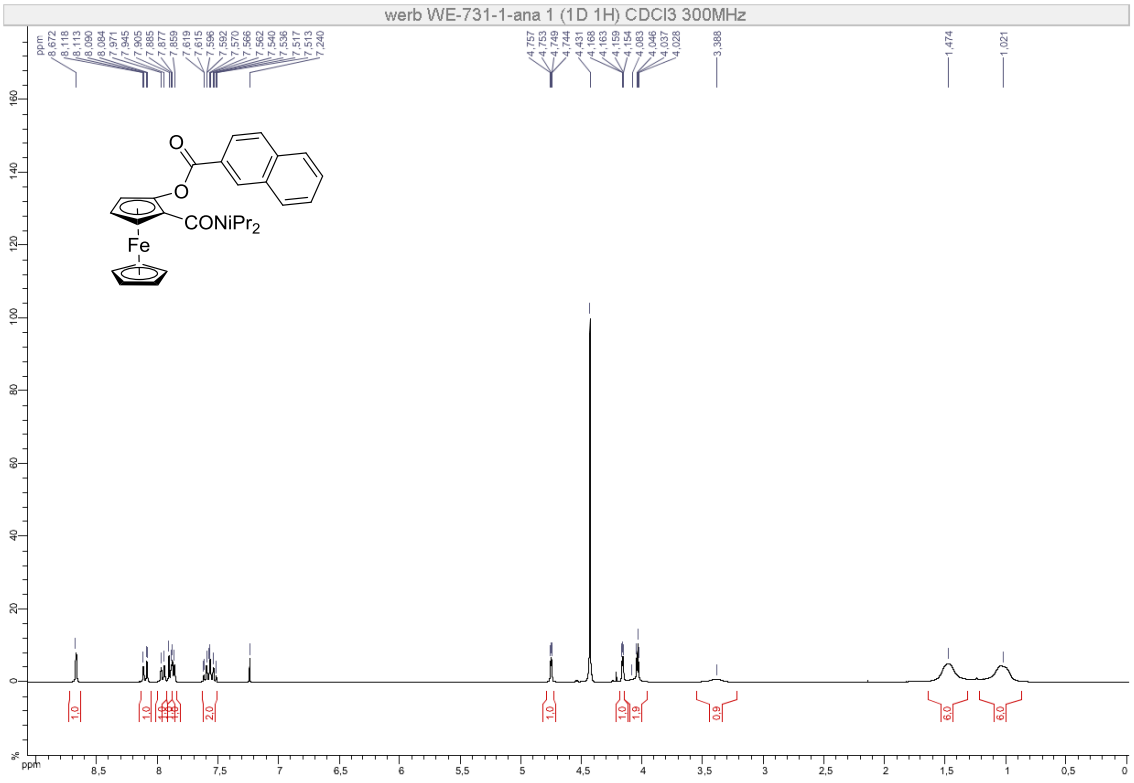

werb WE-720-1-ana 6 (HMBC 1H/13C) CDCl3 300MHz
werb WE-720-1-ana 1 (1D 1H)
O
F
O
CONiPr2
Fe
ppm
werb WE-720-1-ana 2 (1D 13C)

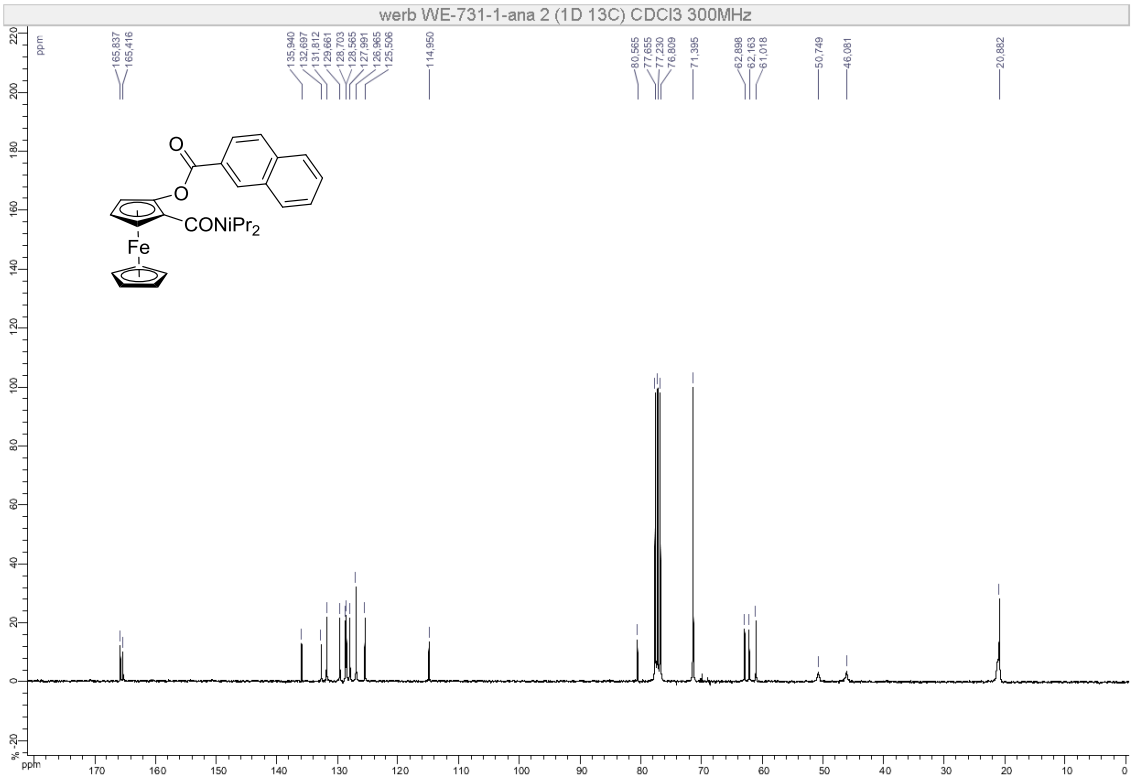


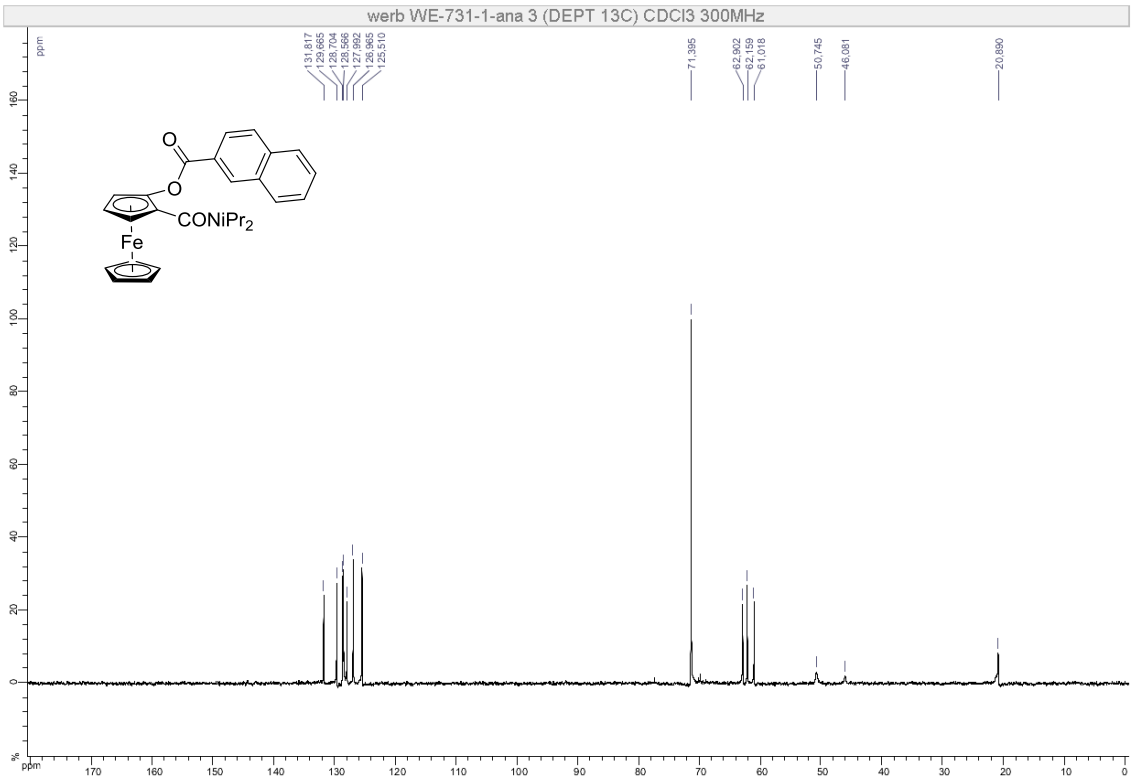
Compound (S_p)-11

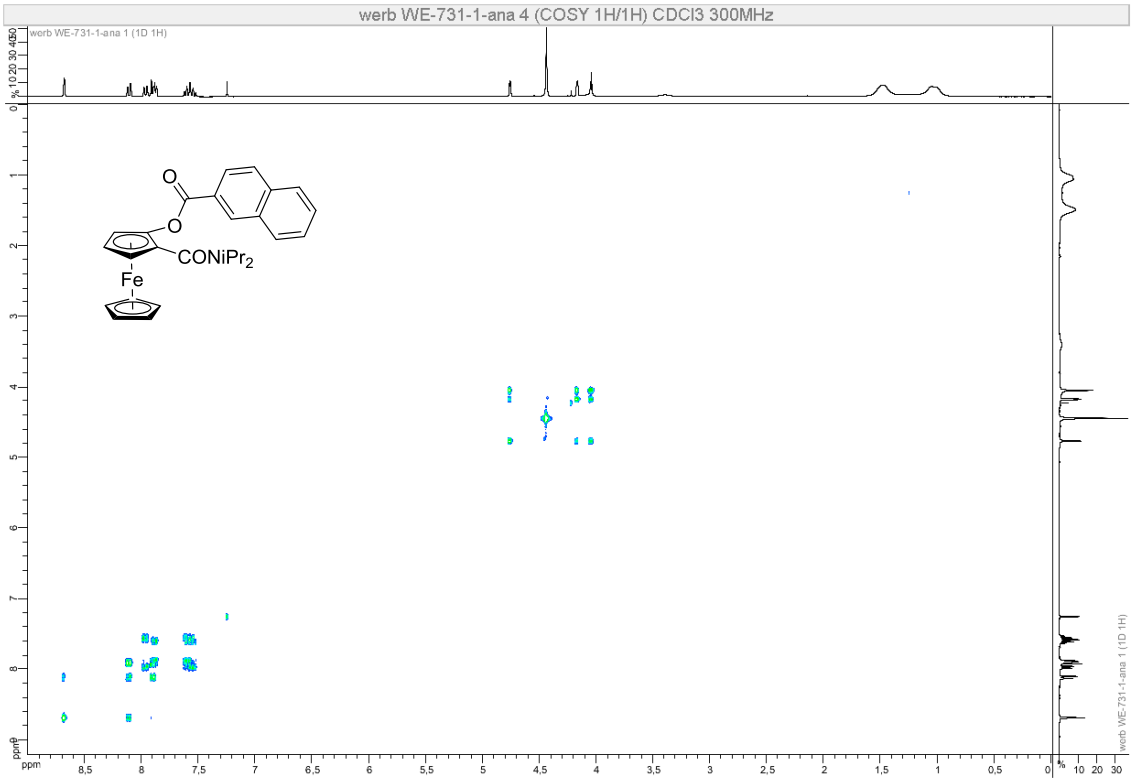


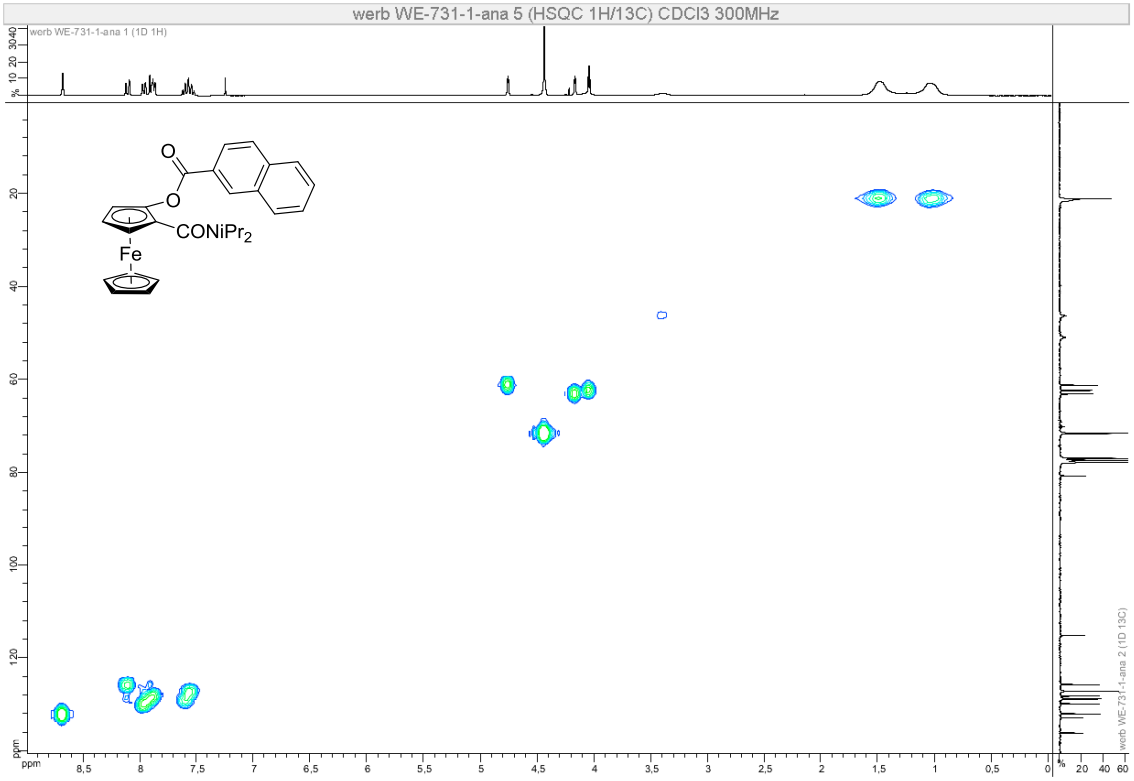
Compound (±)-12

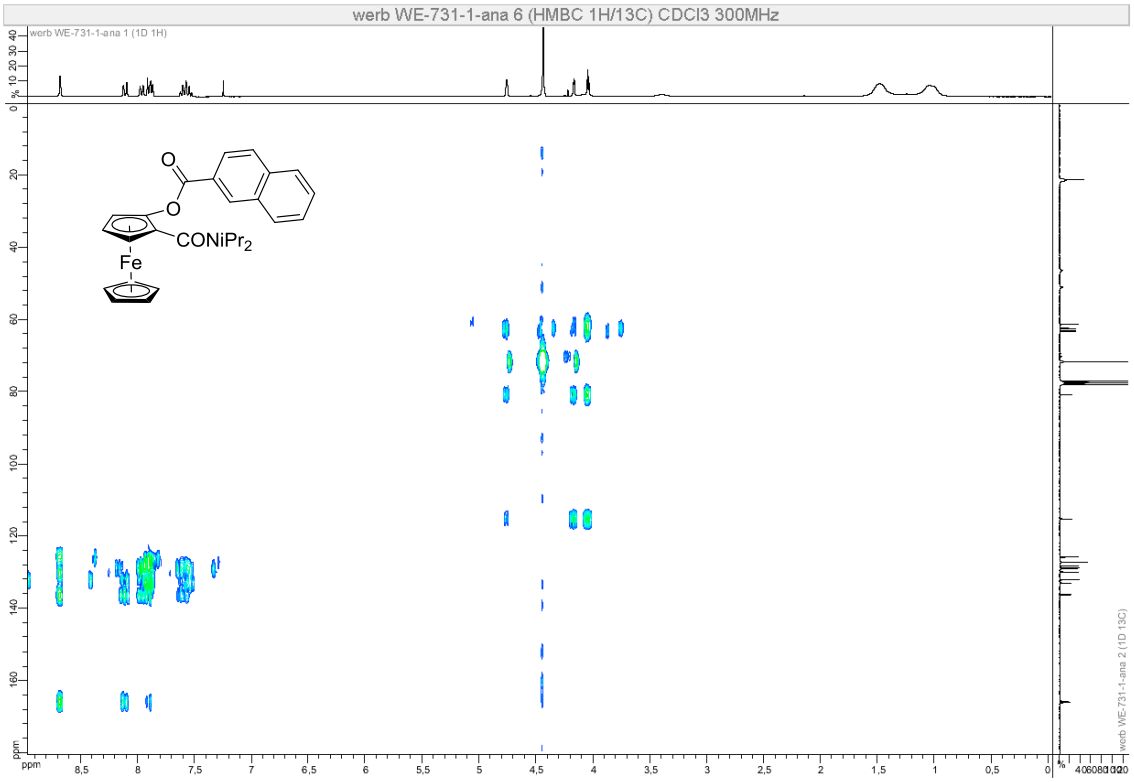

werb WE-731-1-ana 1 (1D 1H) CDCl3 300MHz
O
O
CONiPr2
Fe


werb WE-731-1-ana 2 (1D 13C) CDCl3 300MHz
O
O
CONiPr2
Fe

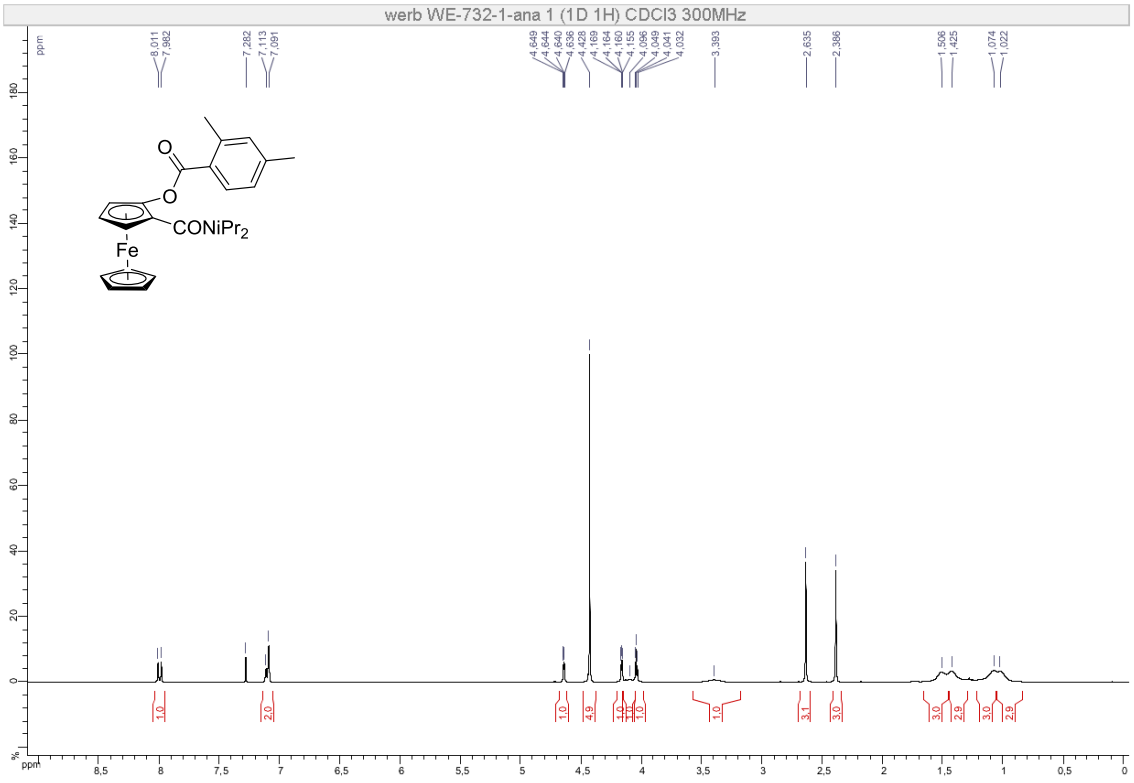

werb WE-731-1-ana 3 (DEPT 13C) CDCl3 300MHz
131,817
130,655
129,704
128,566
127,992
126,965
125,510
71,395
62,902
62,159
61,018
50,745
46,081
20,890
CONiPr2
Fe

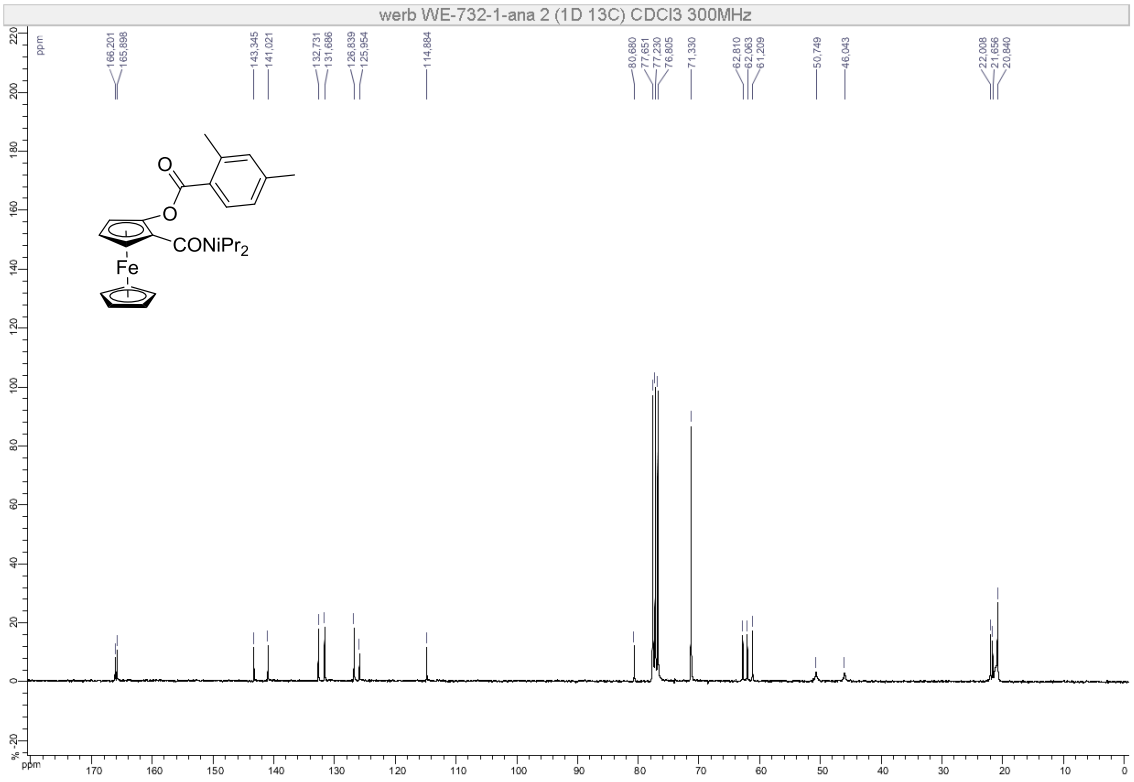

werb WE-731-1-ana 4 (COSY 1H/1H) CDCl3 300MHz
werb WE-731-1-ana 1 (1D 1H)
CONiPr2
Fe

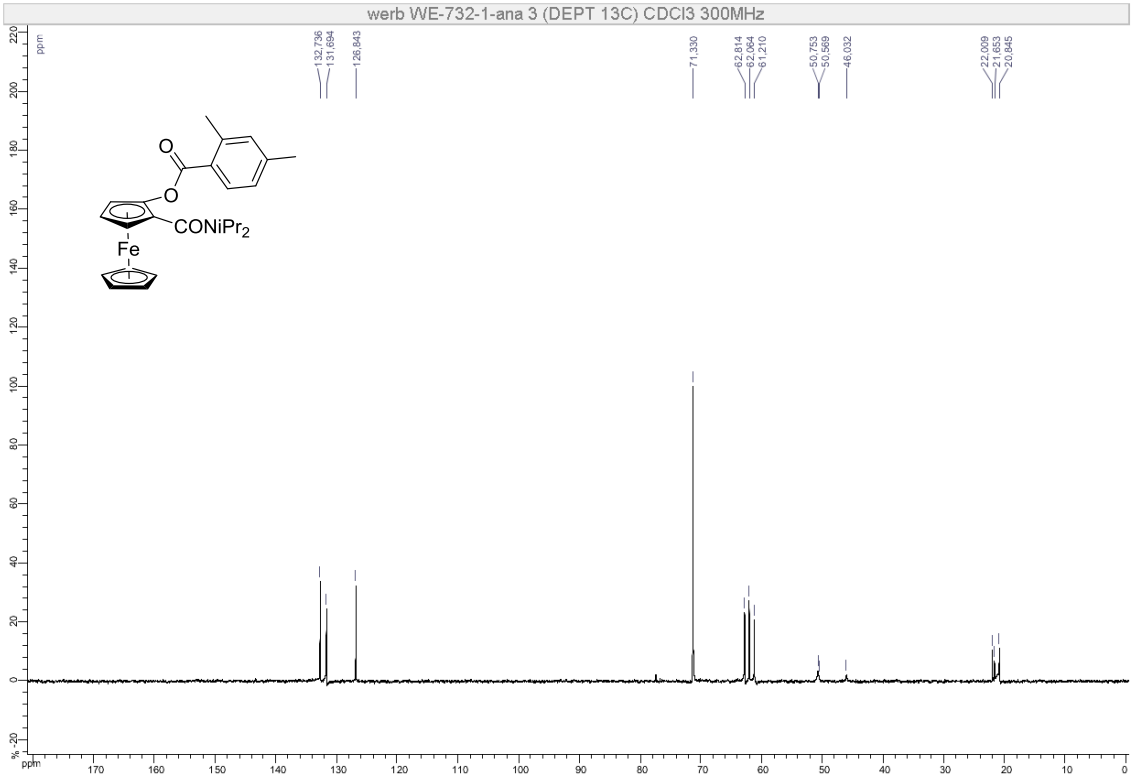

werb WE-731-1-ana 5 (HSQC 1H/13C) CDCl3 300MHz
werb WE-731-1-ana 1 (1D 1H)
O
O
CONiPr2
Fe
ppm
werb WE-731-1-ana 2 (1D 13C)

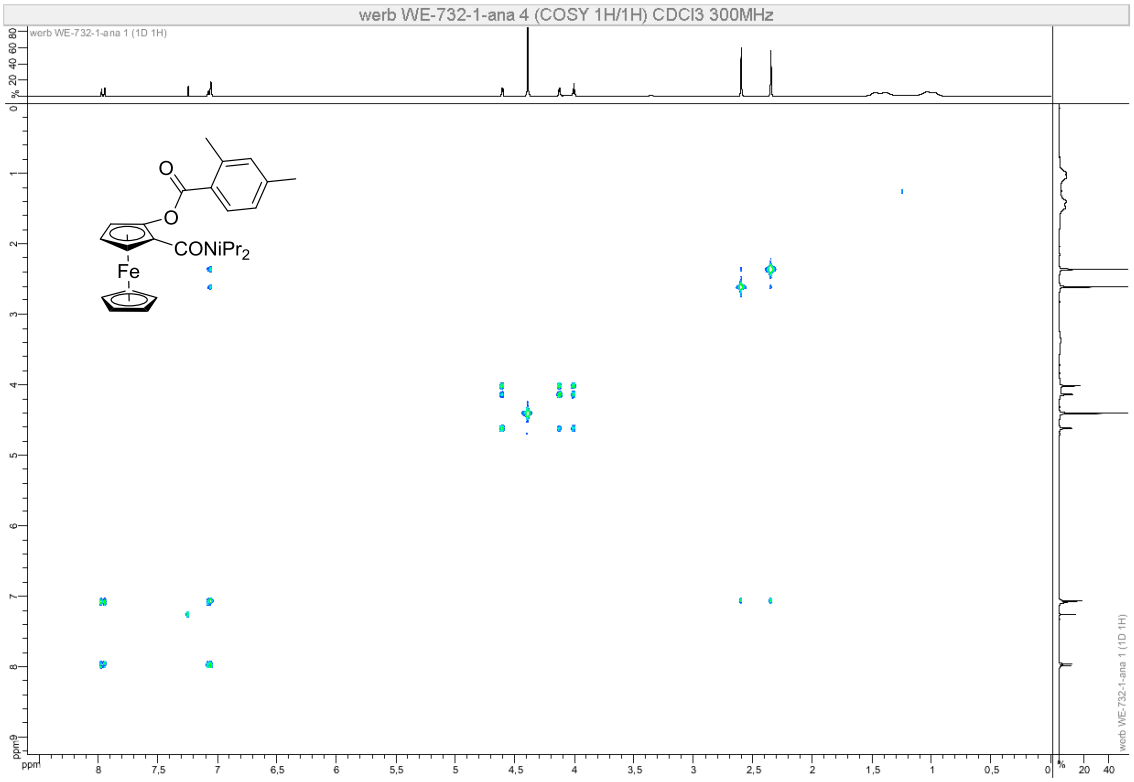

werb WE-731-1-ana 6 (HMBC 1H/13C) CDCl3 300MHz
werb WE-731-1-ana 1 (1D 1H)
O
O
CONiPr2
Fe
ppm
werb WE-731-1-ana 2 (1D 13C)

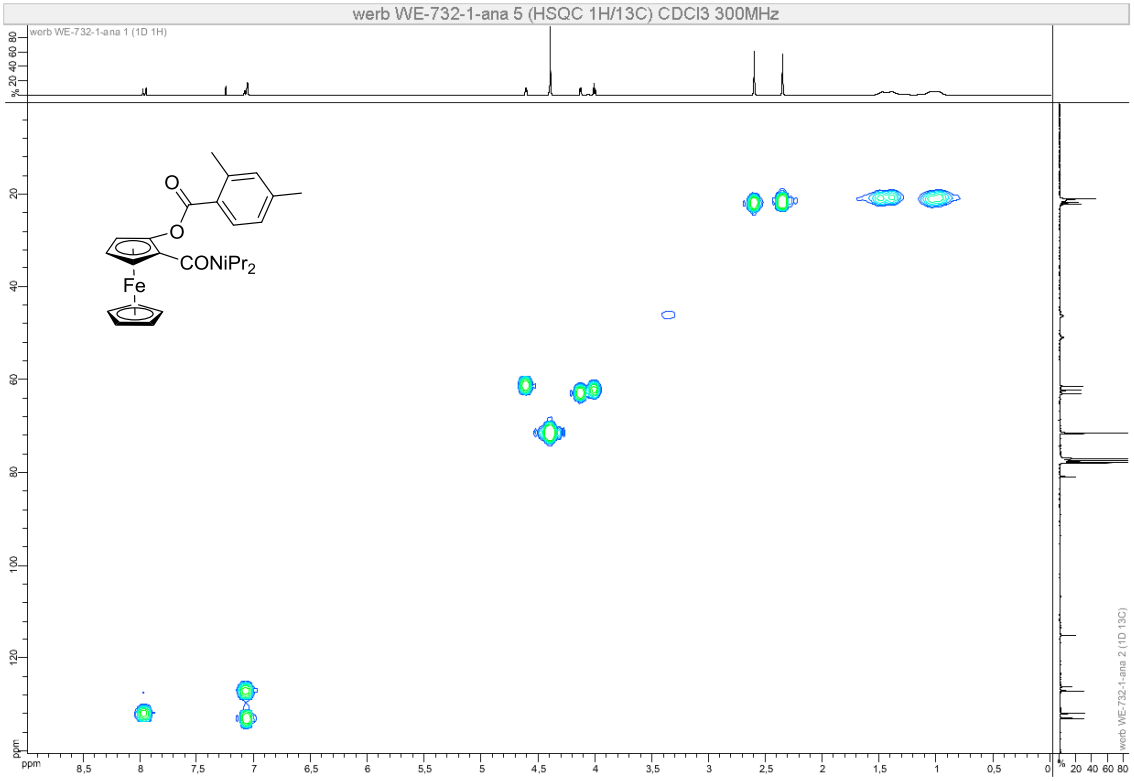
Compound (±)-13

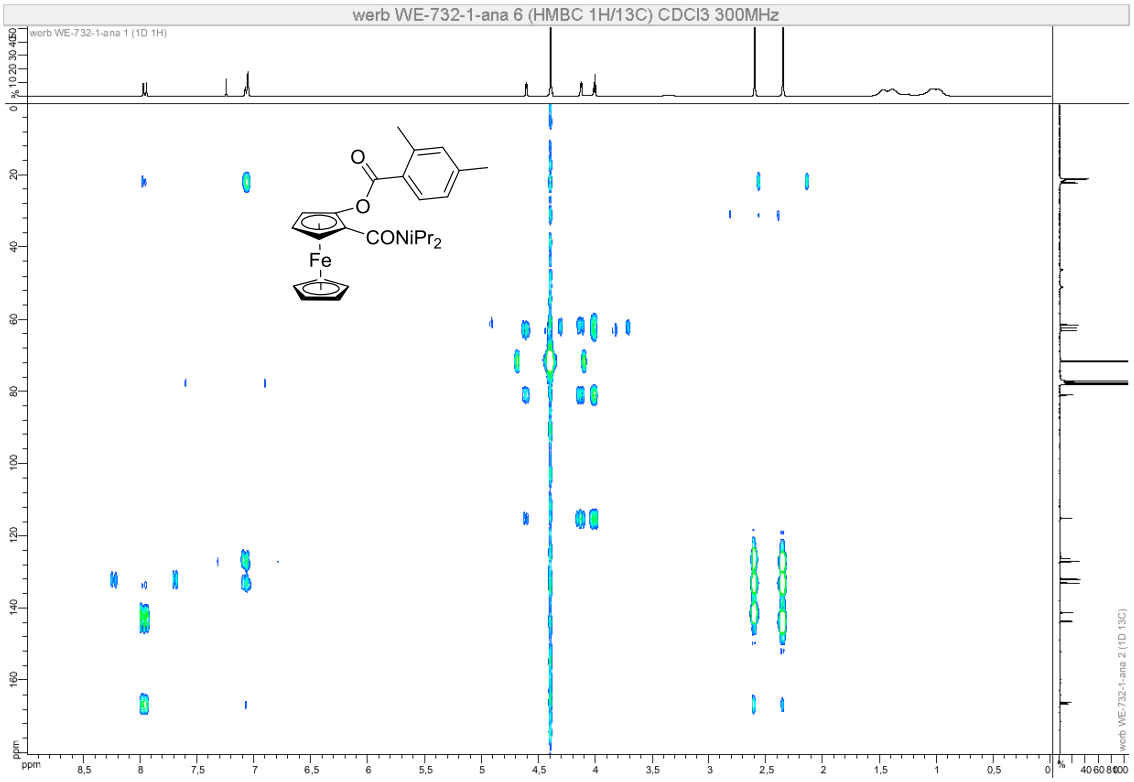

werb WE-732-1-ana 1 (1D 1H) CDCl3 300MHz
O
O
CONiPr2
Fe


werb WE-732-1-ana 2 (1D 13C) CDCl3 300MHz
O
O
CONiPr2
Fe

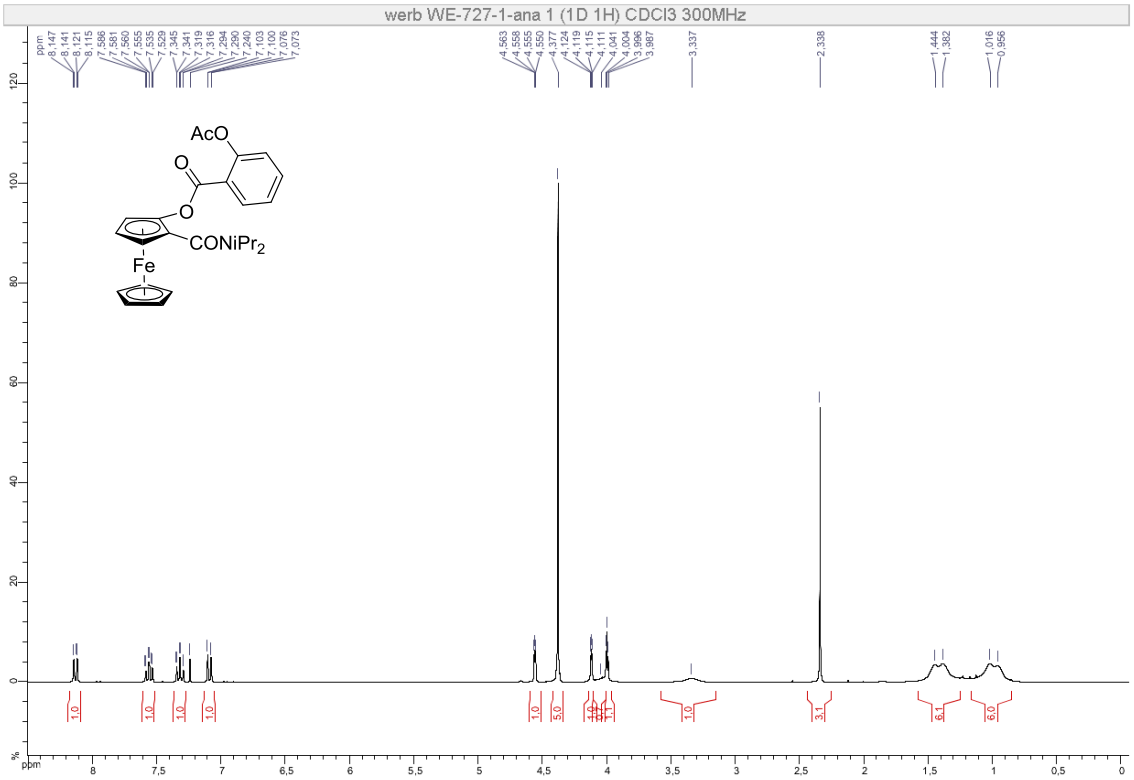

werb WE-732-1-ana 3 (DEPT 13C) CDCl3 300MHz
ppm
132,736
131,694
126,843
71,330
62,814
62,064
61,210
50,753
50,569
46,032
22,009
21,653
20,845
O
O
CONiPr2
Fe

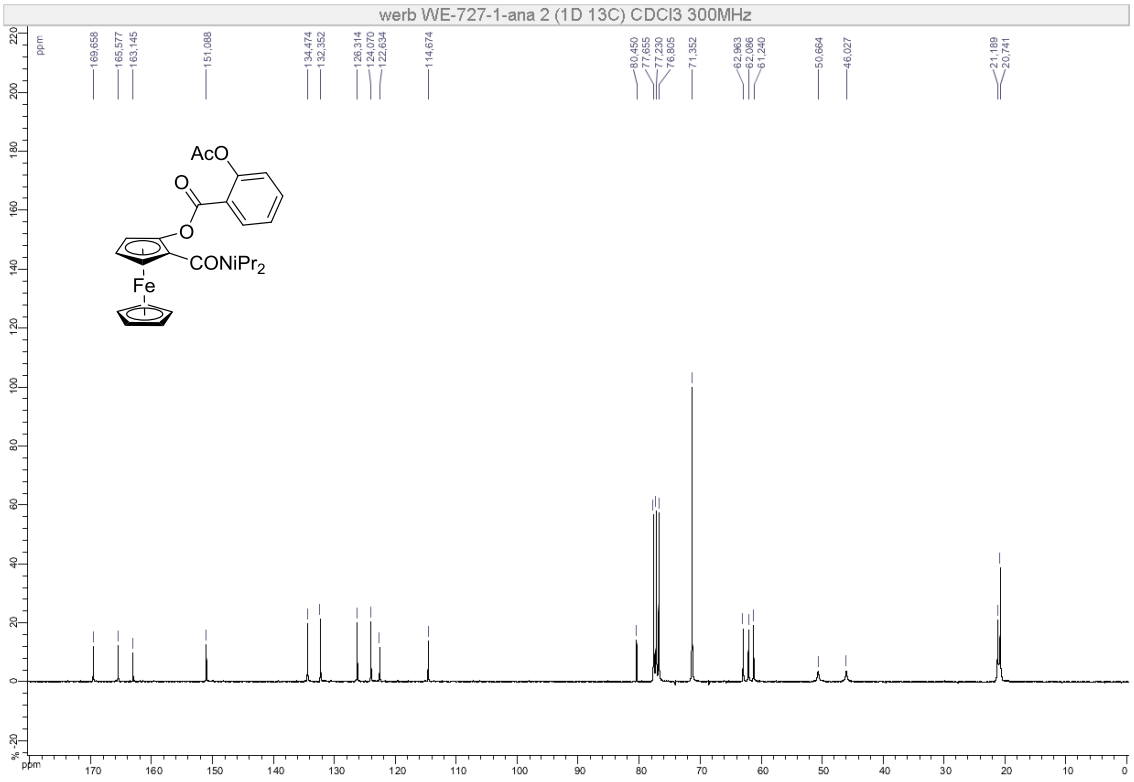

werb WE-732-1-ana 4 (COSY 1H/1H) CDCl3 300MHz
werb WE-732-1-ana 1 (1D 1H)
O
O
CONiPr2
Fe
ppm

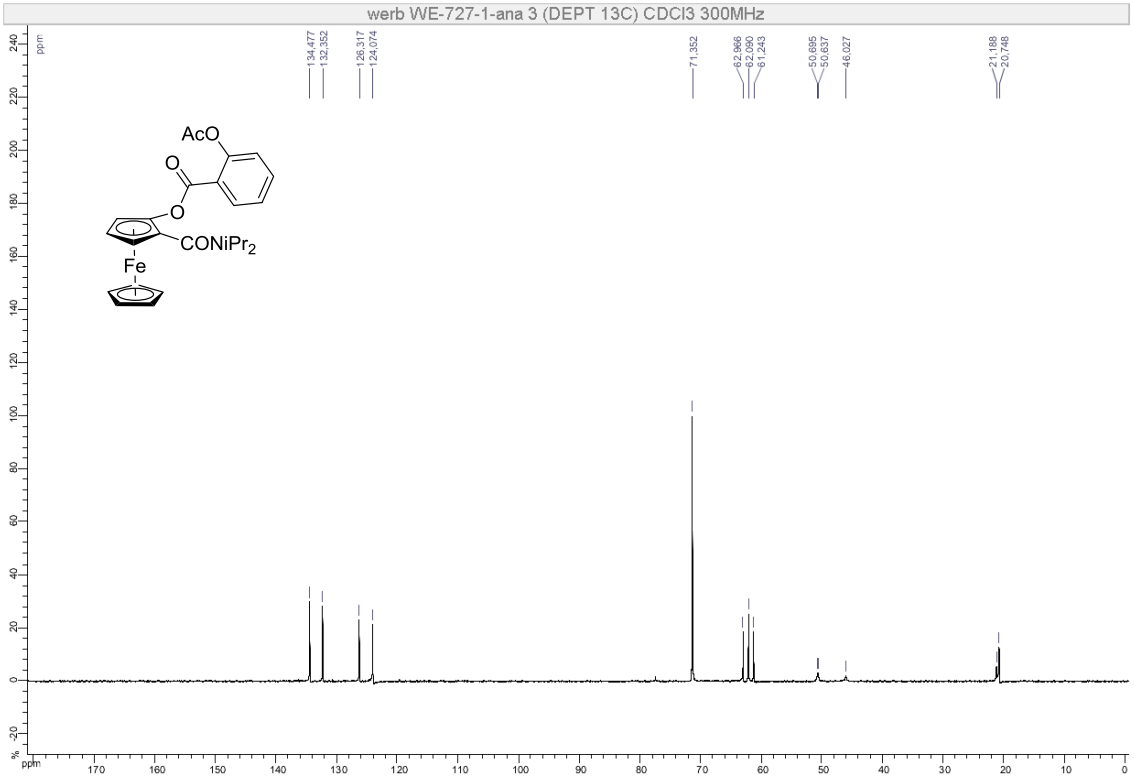

werb WE-732-1-ana 5 (HSQC 1H/13C) CDCl3 300MHz
werb WE-732-1-ana 1 (1D 1H)
werb WE-732-1-ana 2 (1D 13C)
ppm
O
O
CONiPr2
Fe

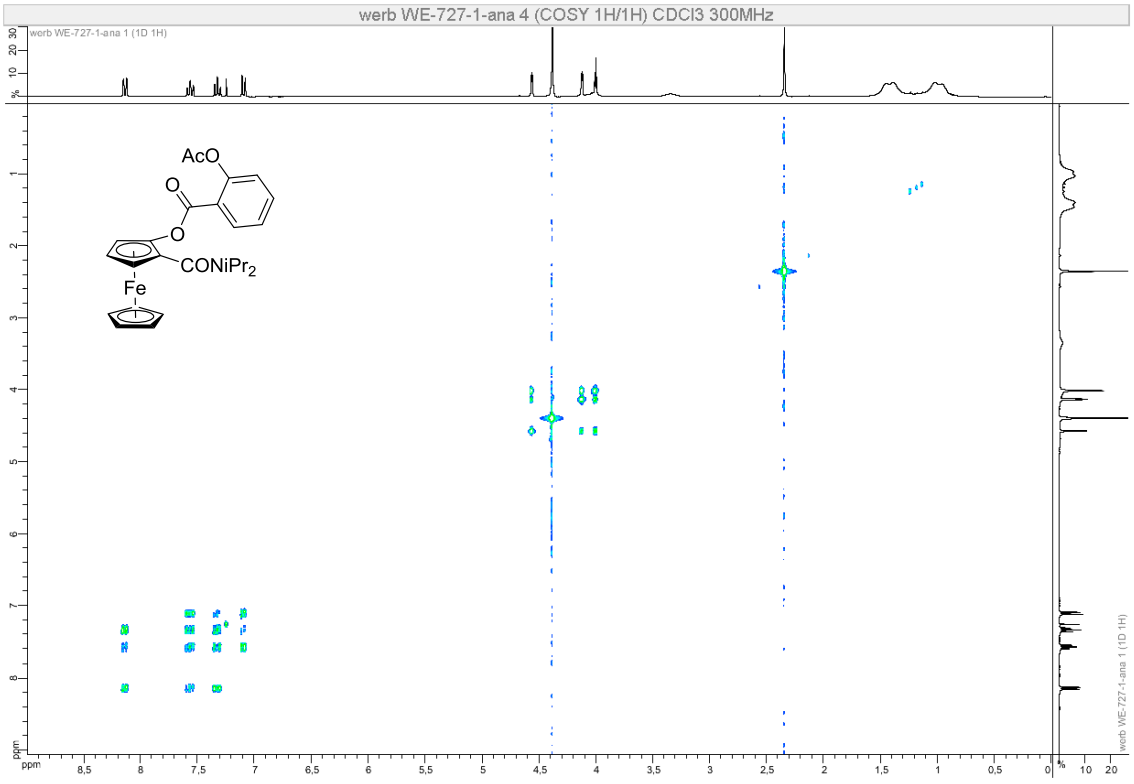

werb WE-732-1-ana 6 (HMBC 1H/13C) CDCl3 300MHz
werb WE-732-1-ana 1 (1D 1H)
werb WE-732-1-ana 2 (1D 13C)
ppm
O
O
CONiPr2
Fe

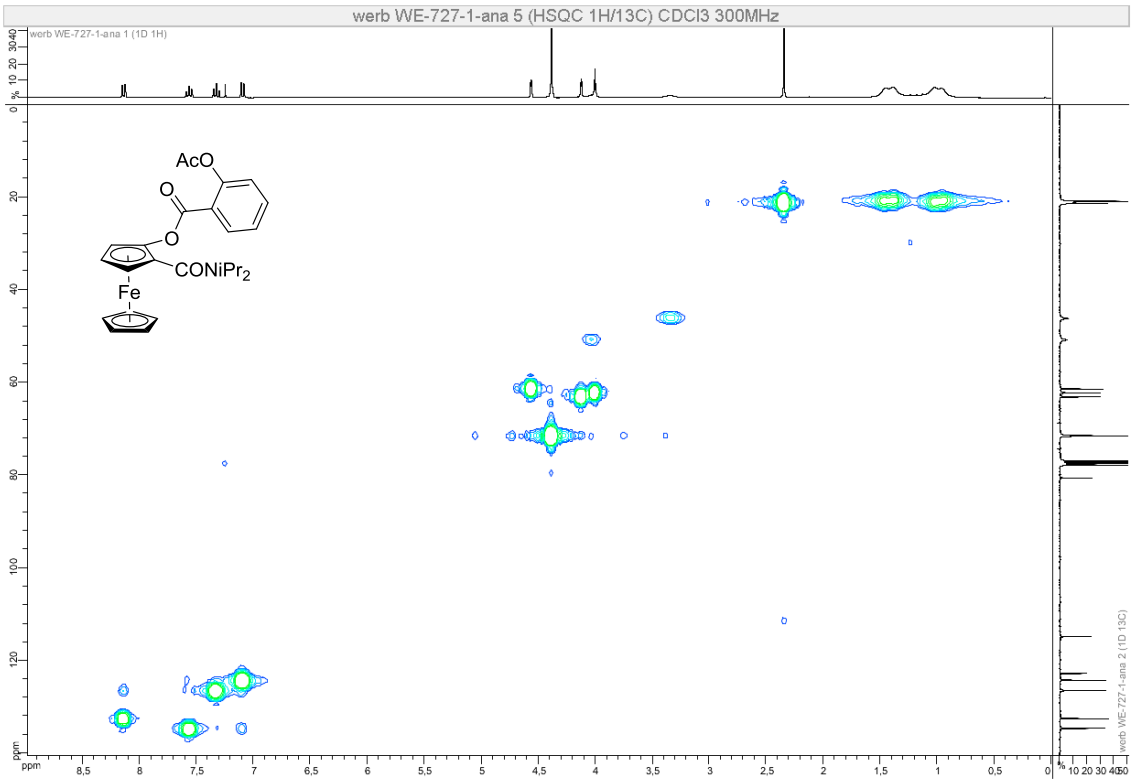
Compound (±)-14

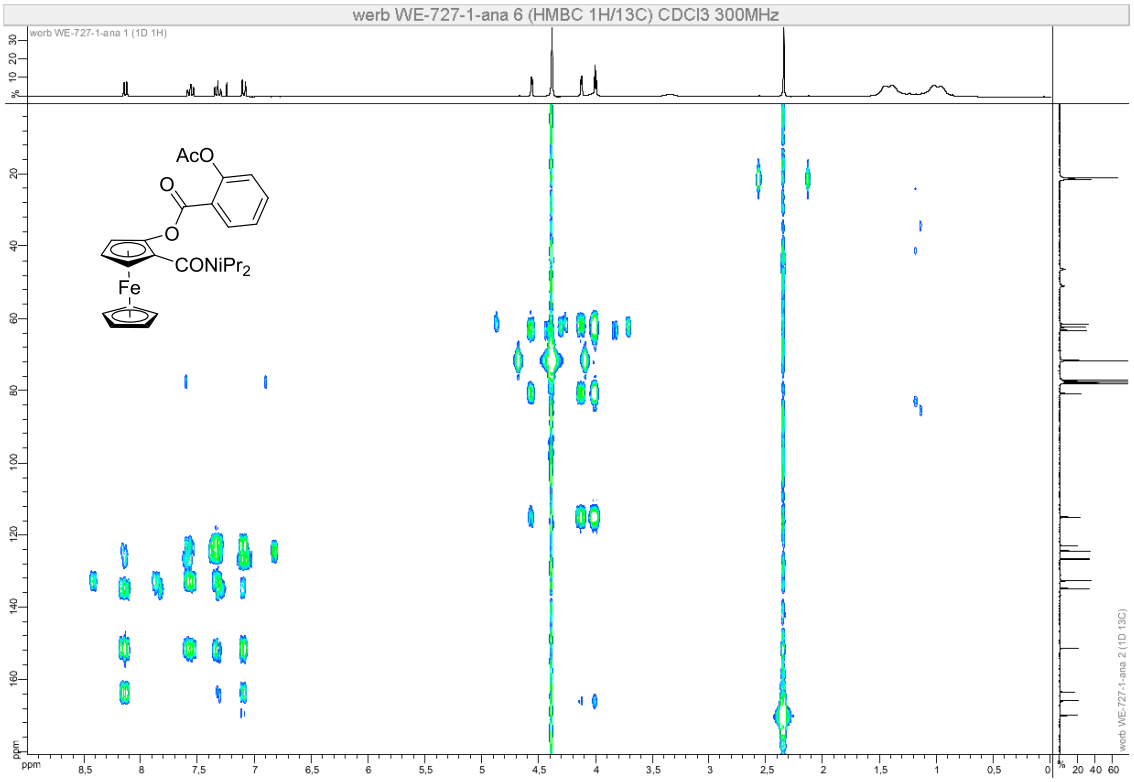

werb WE-727-1-ana 1 (1D 1H) CDCl3 300MHz
AcO
O
O
CONiPr2
Fe


werb WE-727-1-ana 2 (1D 13C) CDCl3 300MHz
AcO
O
O
CONiPr2
Fe

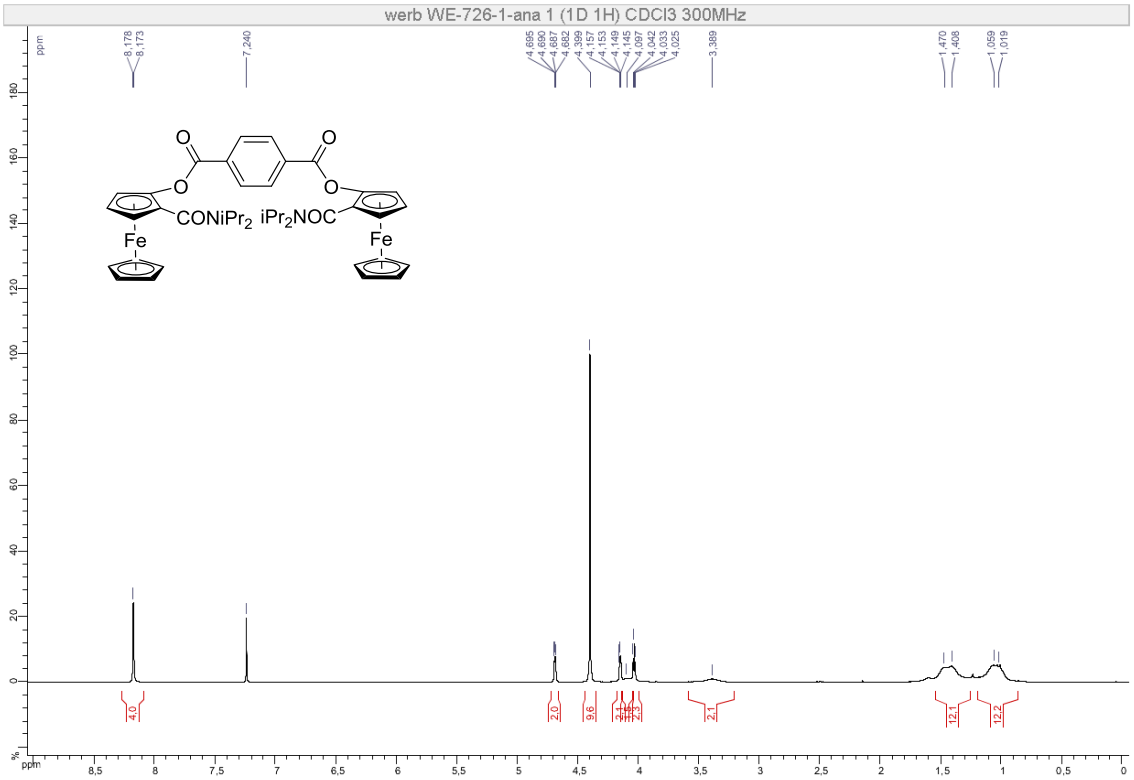

werb WE-727-1-ana 3 (DEPT 13C) CDCl3 300MHz
134,477
132,352
126,317
124,074
71,352
62,966
62,090
61,243
50,685
50,637
46,027
21,188
20,748
AcO
O
O
CONiPr2
Fe
ppm

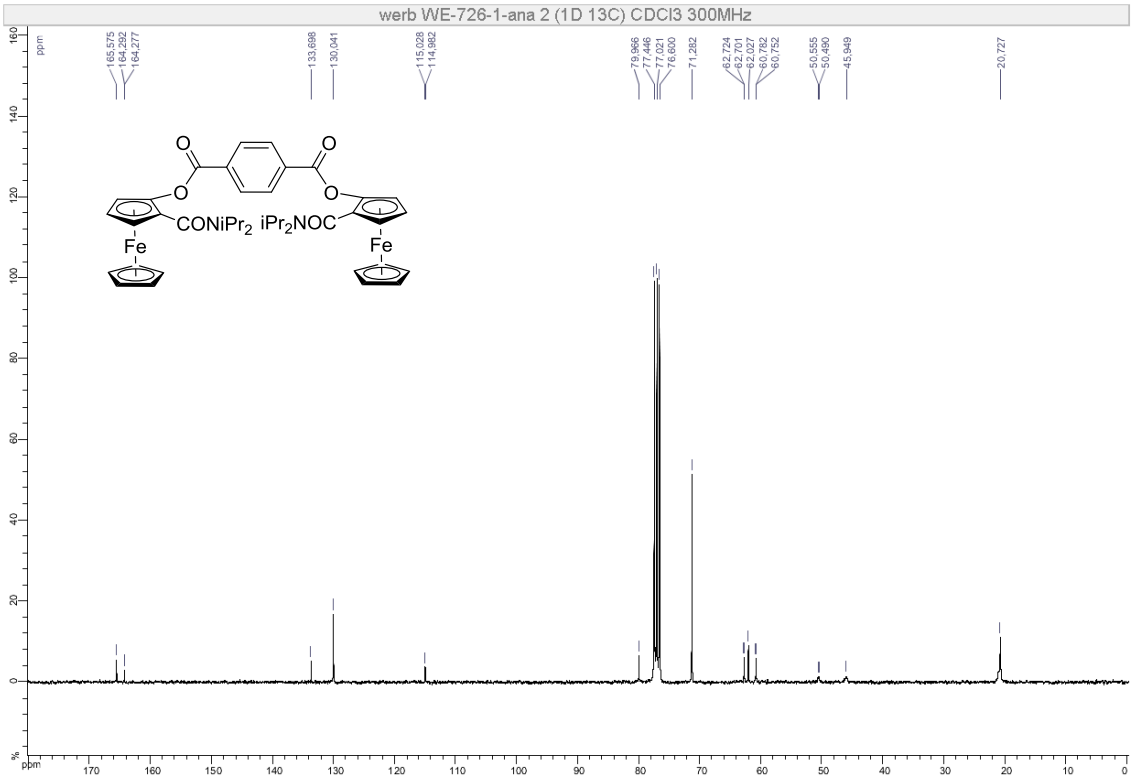

werb WE-727-1-ana 4 (COSY 1H/1H) CDCl3 300MHz
werb WE-727-1-ana 1 (1D 1H)
AcO
O
O
CONiPr2
Fe
ppm
werb WE-727-1-ana 1 (1D 1H)

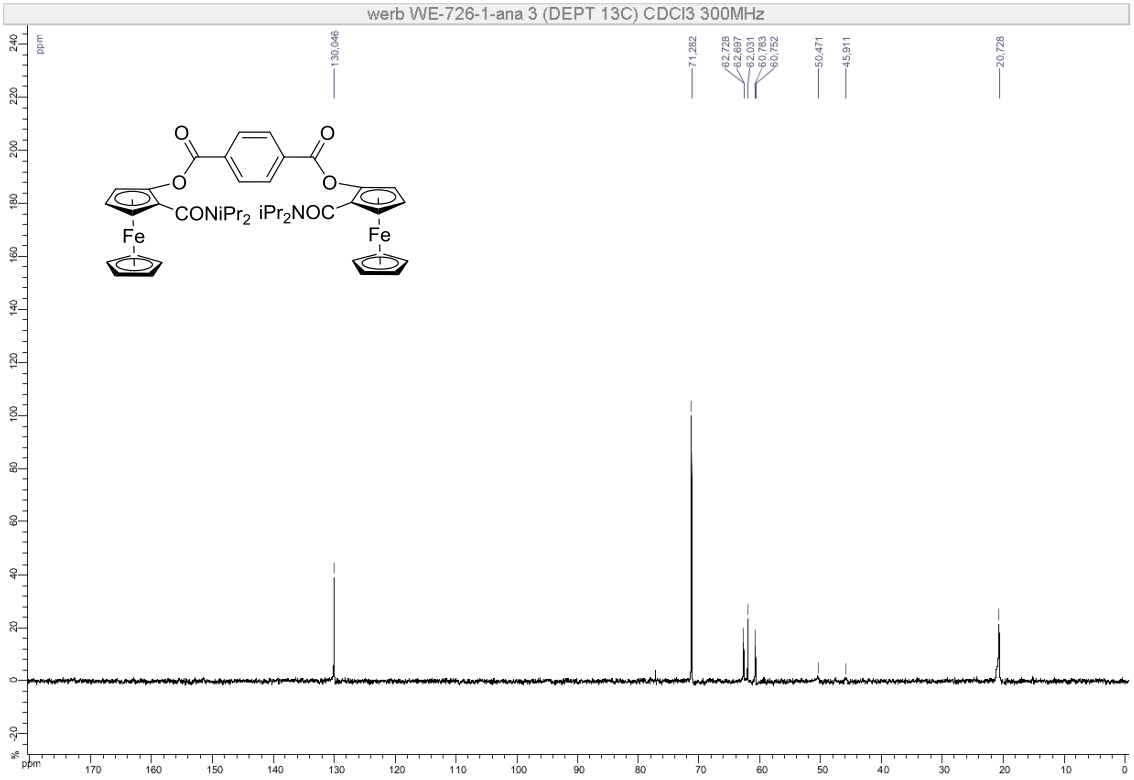

werb WE-727-1-ana 5 (HSQC 1H/13C) CDCl3 300MHz
werb WE-727-1-ana 1 (1D 1H)
AcO
O
O
CONiPr2
Fe
ppm
werb WE-727-1-ana 2 (1D 13C)

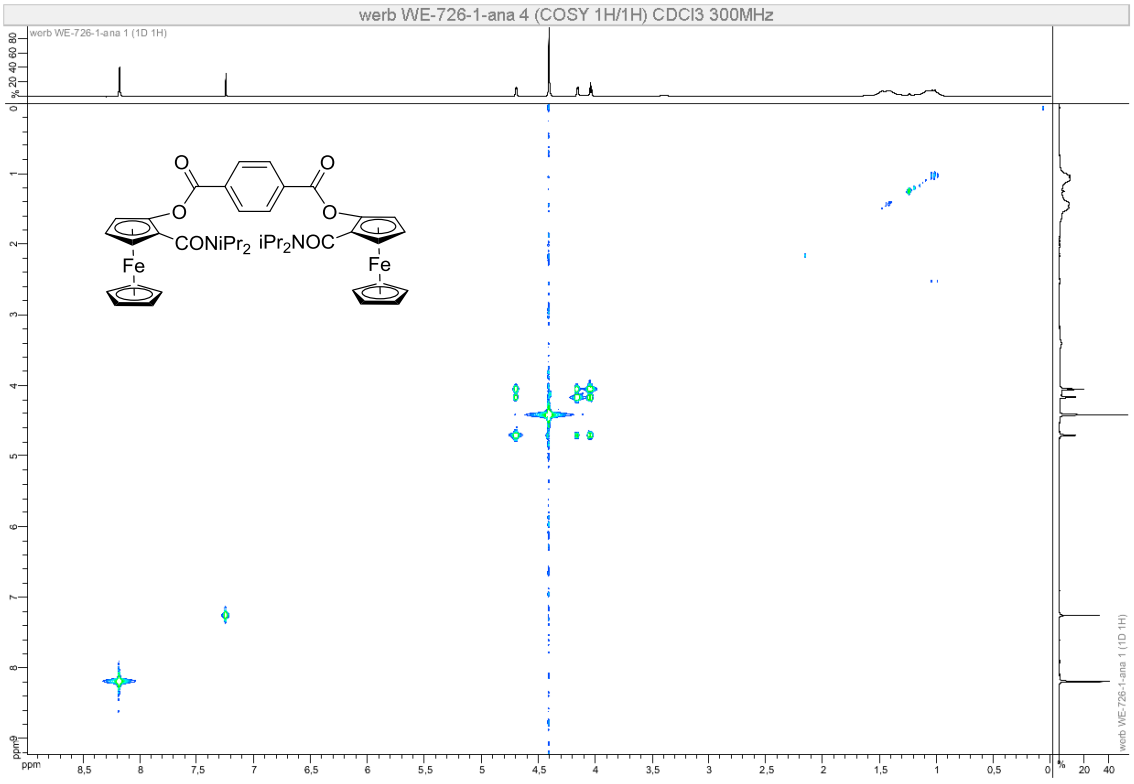

werb WE-727-1-ana 6 (HMBC 1H/13C) CDCl3 300MHz
werb WE-727-1-ana 1 (1D 1H)
AcO
O
O
CONiPr2
Fe
ppm
werb WE-727-1-ana 2 (1D 13C)

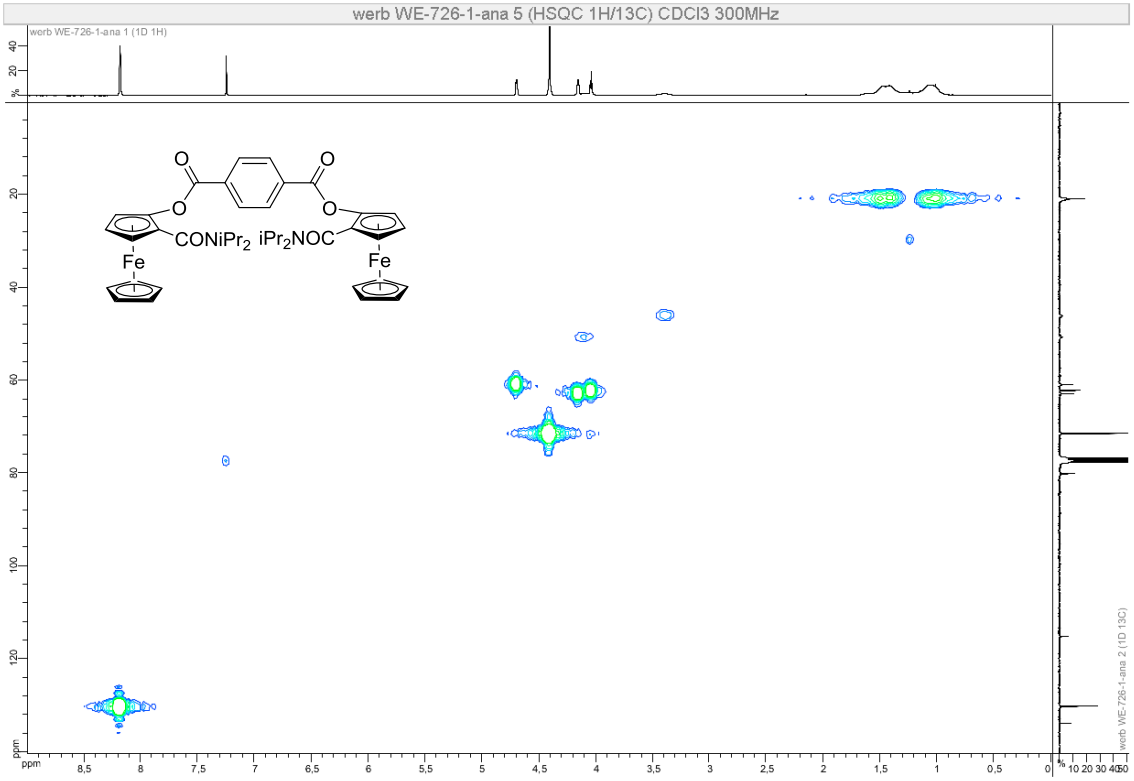
Compound (±)-15

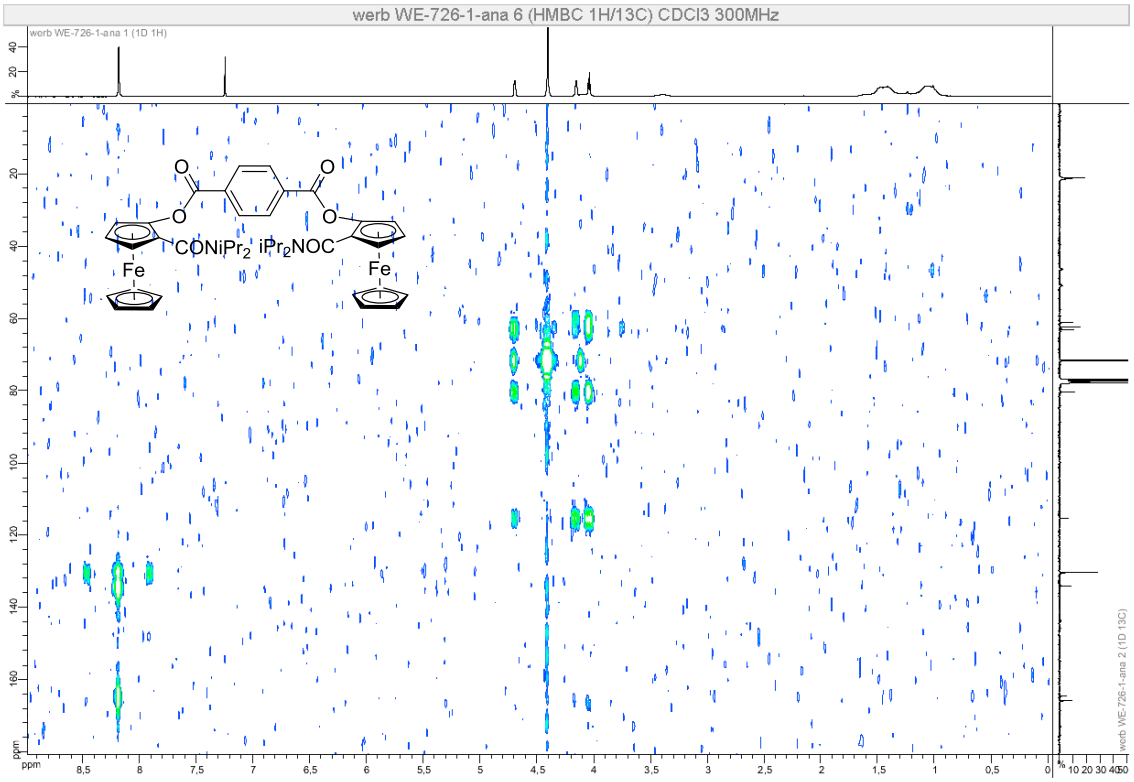

werb WE-726-1-ana 1 (1D 1H) CDCl3 300MHz
CONiPr2 iPr2NOC
Fe
Fe


werb WE-726-1-ana 2 (1D 13C) CDCl3 300MHz
CONiPr2 iPr2NOC
Fe
Fe

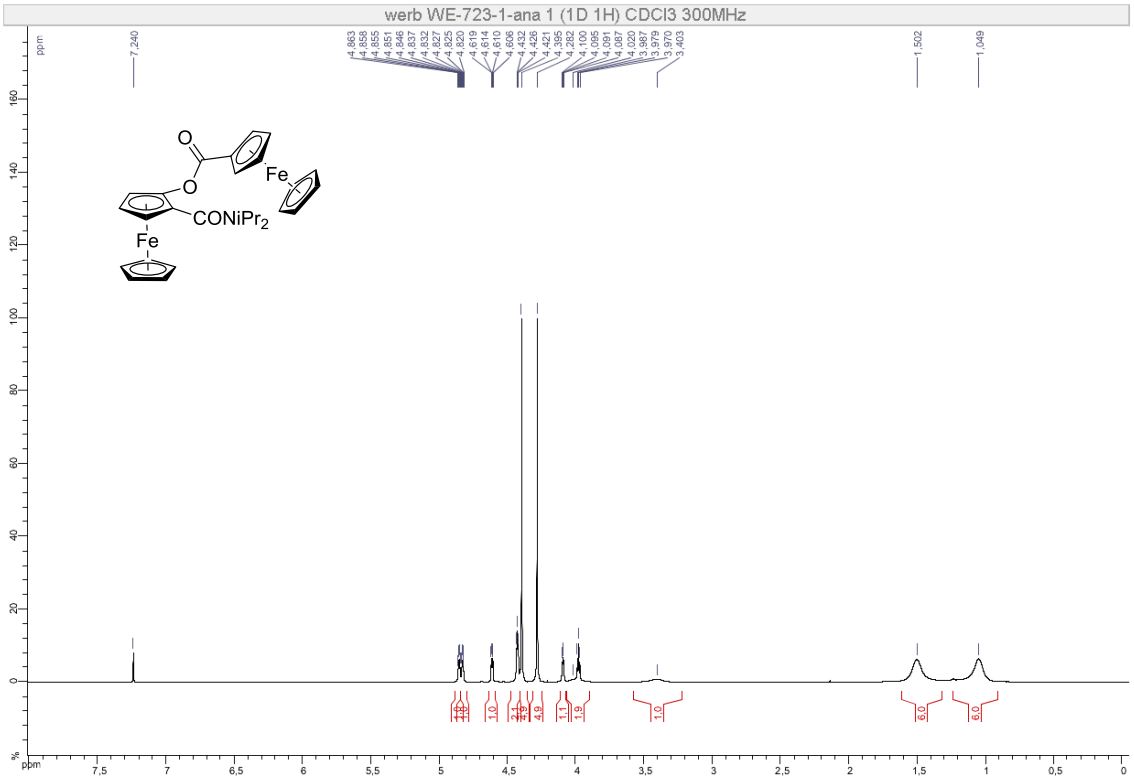

werb WE-726-1-ana 3 (DEPT 13C) CDCl3 300MHz
130,046
71,282
62,728
62,697
62,031
60,783
60,752
50,471
45,911
20,728
CONiPr2 iPr2NOC
Fe
Fe

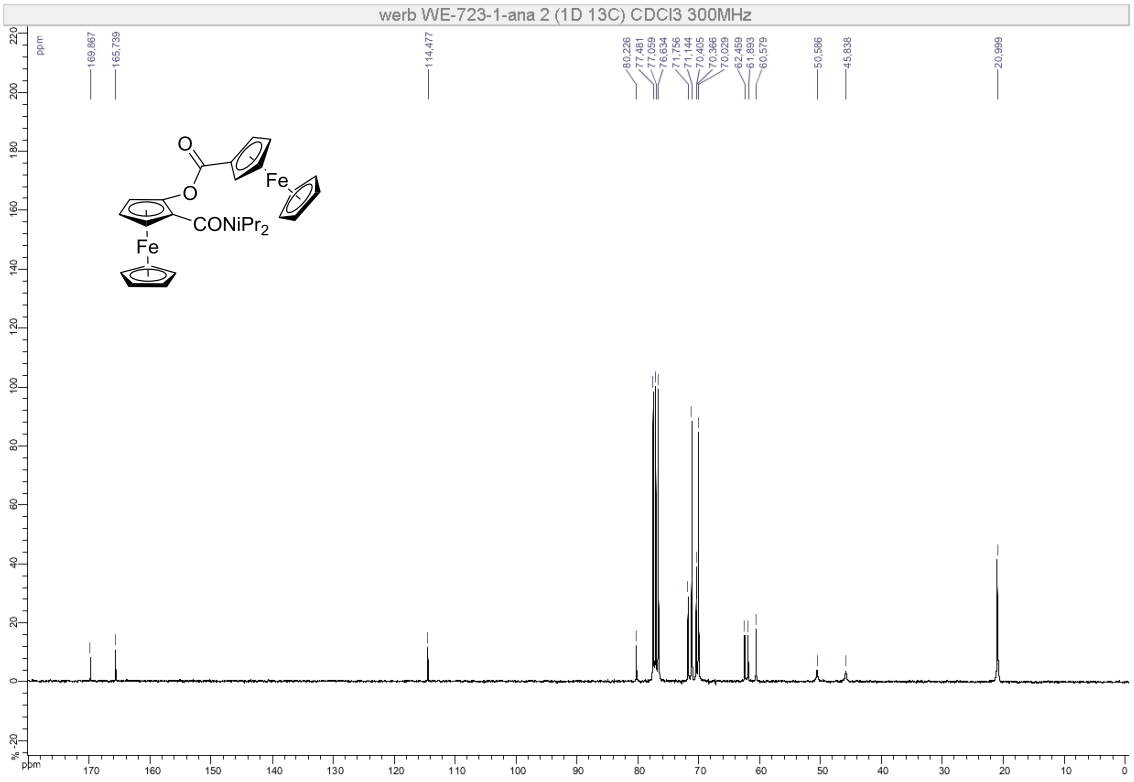

werb WE-726-1-ana 4 (COSY 1H/1H) CDCl3 300MHz
werb WE-726-1-ana 1 (1D 1H)
CONiPr2 iPr2NOC
Fe
Fe

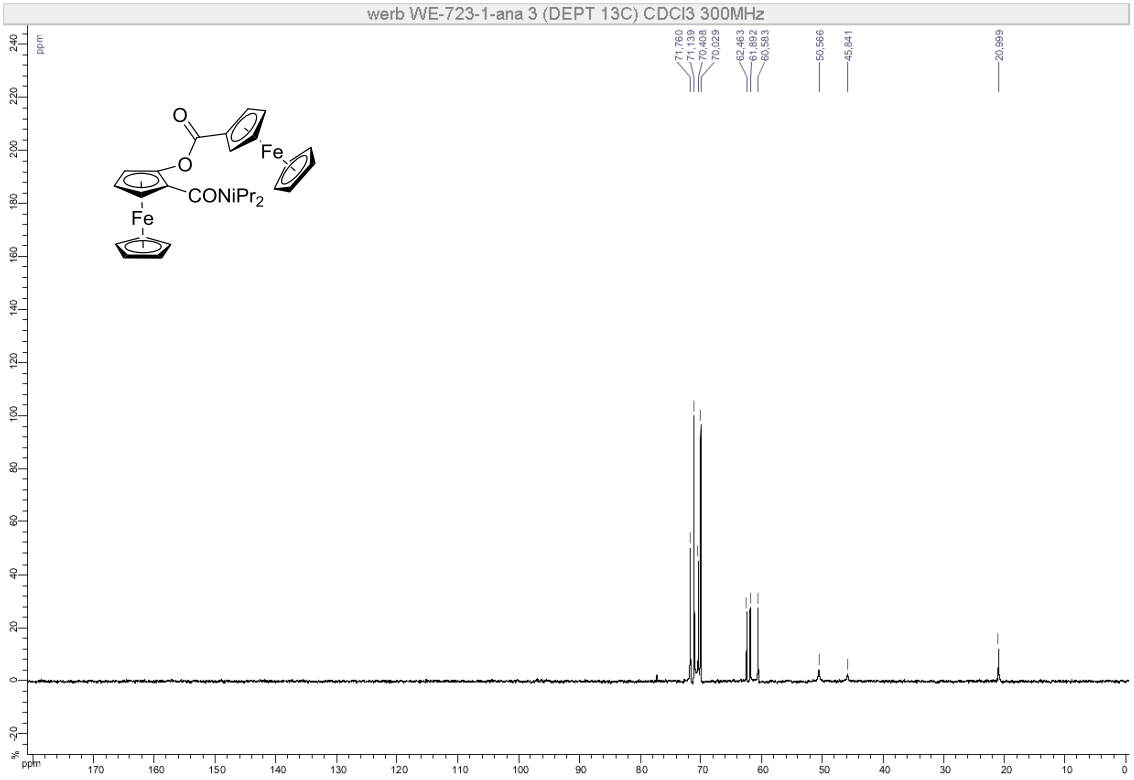

werb WE-726-1-ana 5 (HSQC 1H/13C) CDCl3 300MHz
werb WE-726-1-ana 1 (1D 1H)
CONiPr2 iPr2NOC
Fe
Fe
ppm
werb WE-726-1-ana 2 (1D 13C)

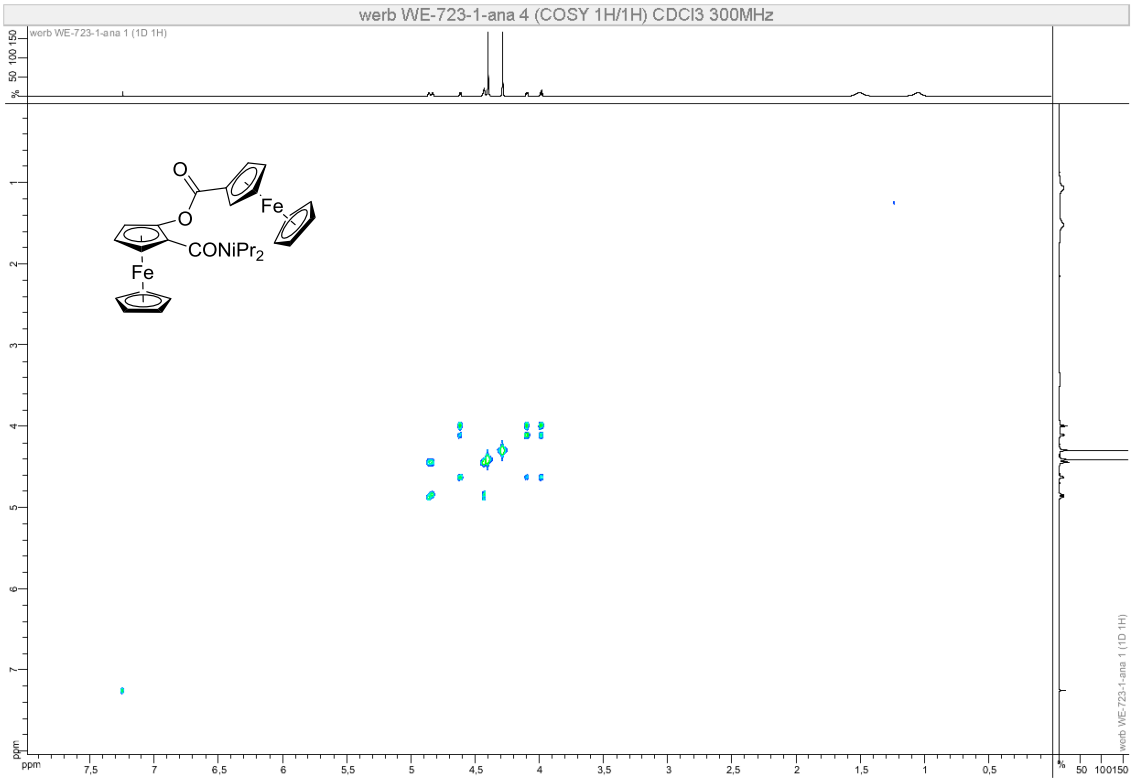

werb WE-726-1-ana 6 (HMBC 1H/13C) CDCl3 300MHz
werb WE-726-1-ana 1 (1D 1H)
CONiPr2 iPr2NOC
Fe
Fe
ppm
werb WE-726-1-ana 2 (1D 13C)

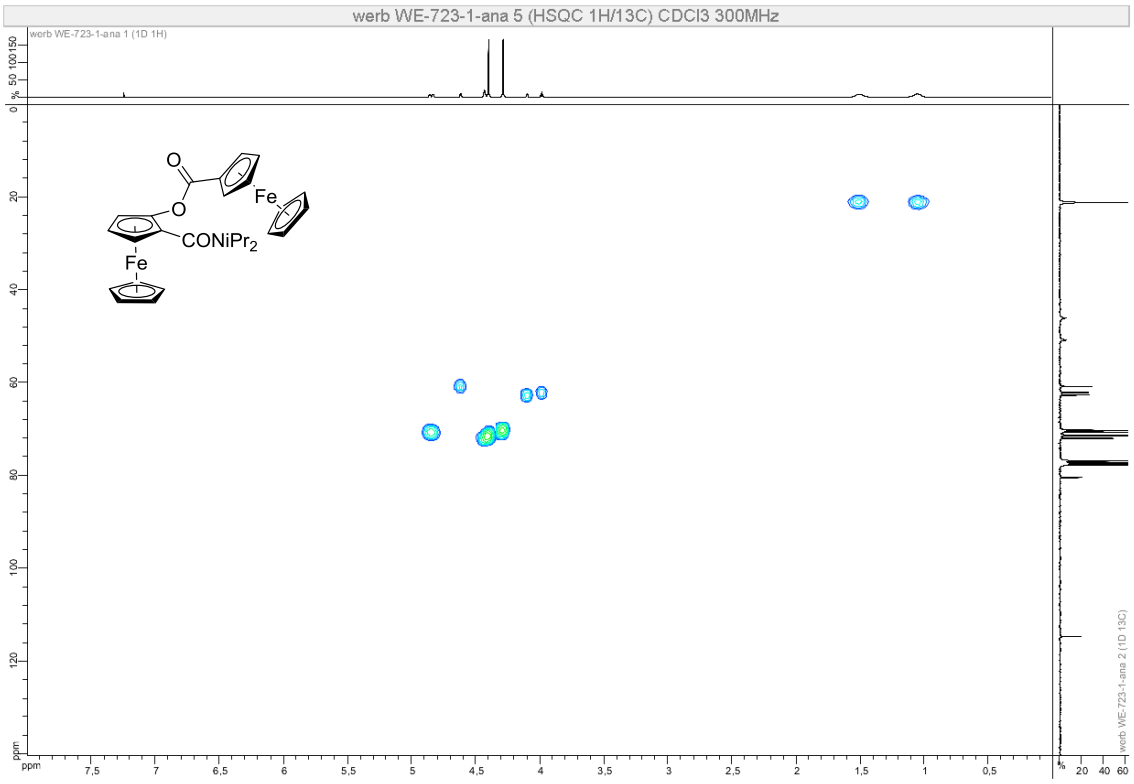
Compound (±)-16

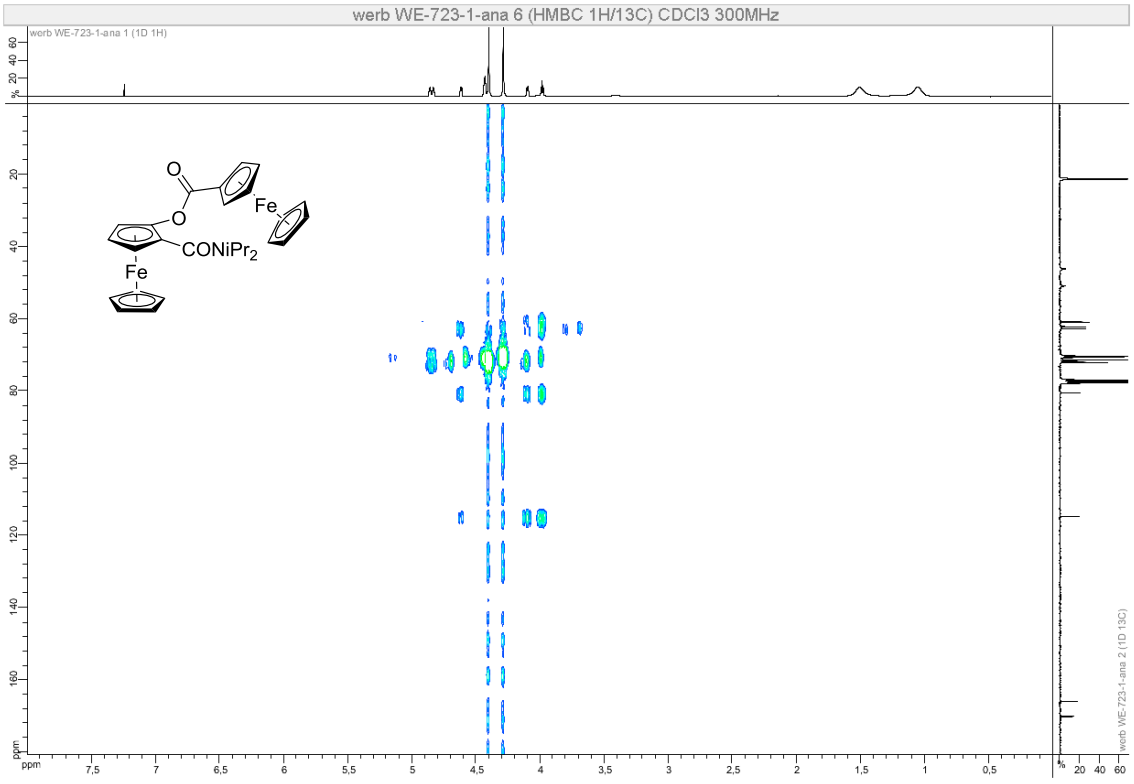

werb WE-723-1-ana 1 (1D 1H) CDCl3 300MHz
O
O
Fe
CONiPr2
Fe


werb WE-723-1-ana 2 (1D 13C) CDCl3 300MHz
O
O
Fe
CONiPr2
Fe

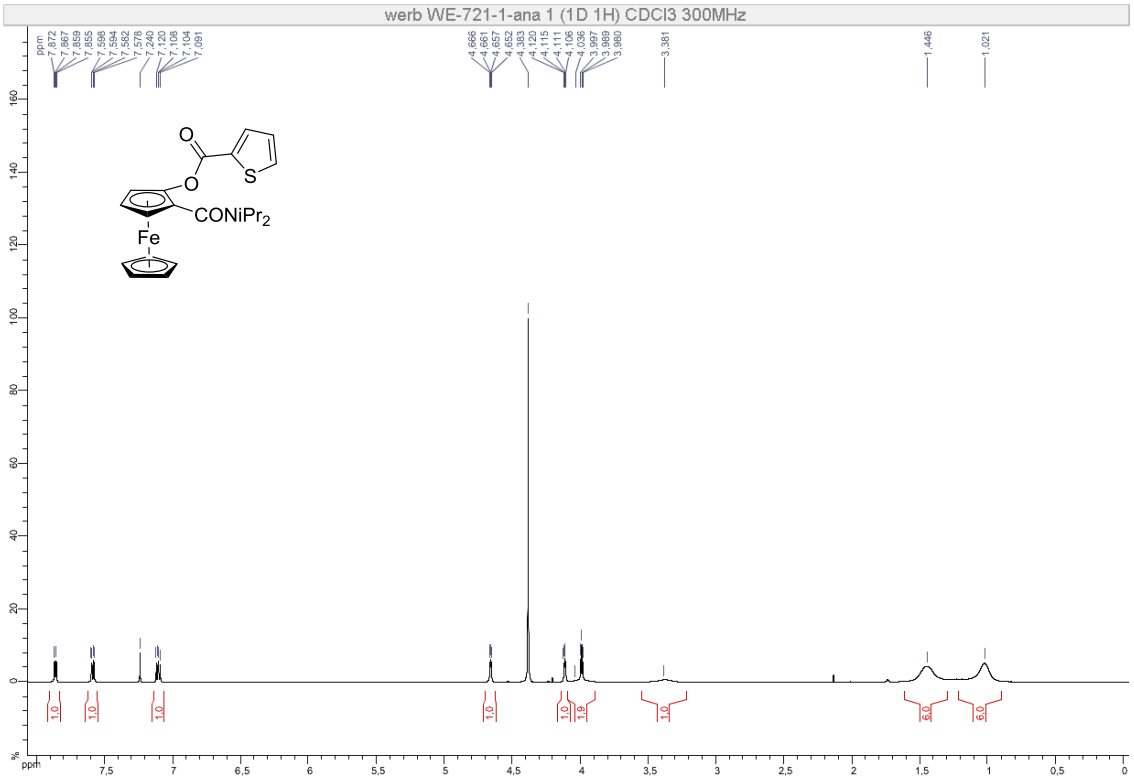

werb WE-723-1-ana 3 (DEPT 13C) CDCl3 300MHz
71,760
71,139
70,408
70,029
62,463
61,892
60,583
50,566
45,841
20,999
O
O
Fe
CONiPr2
Fe

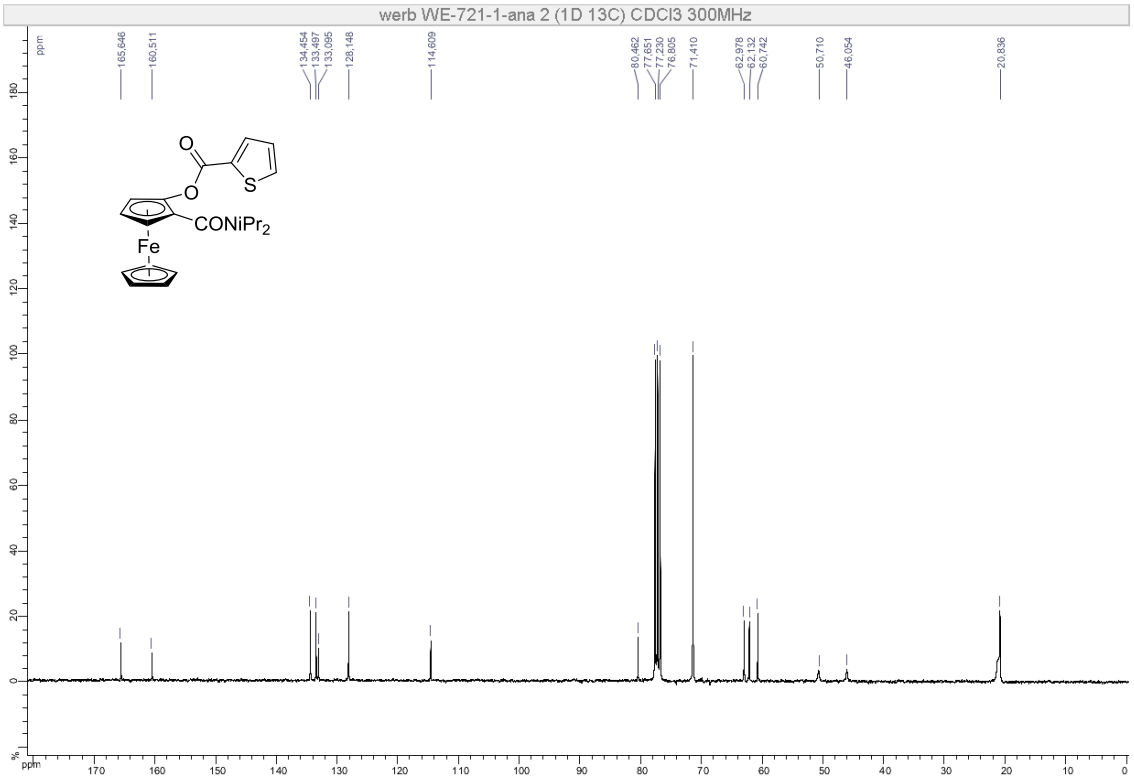

werb WE-723-1-ana 4 (COSY 1H/1H) CDCl3 300MHz
werb WE-723-1-ana 1 (1D 1H)
O
O
Fe
CONiPr2
Fe
ppm
werb WE-723-1-ana 1 (1D 1H)

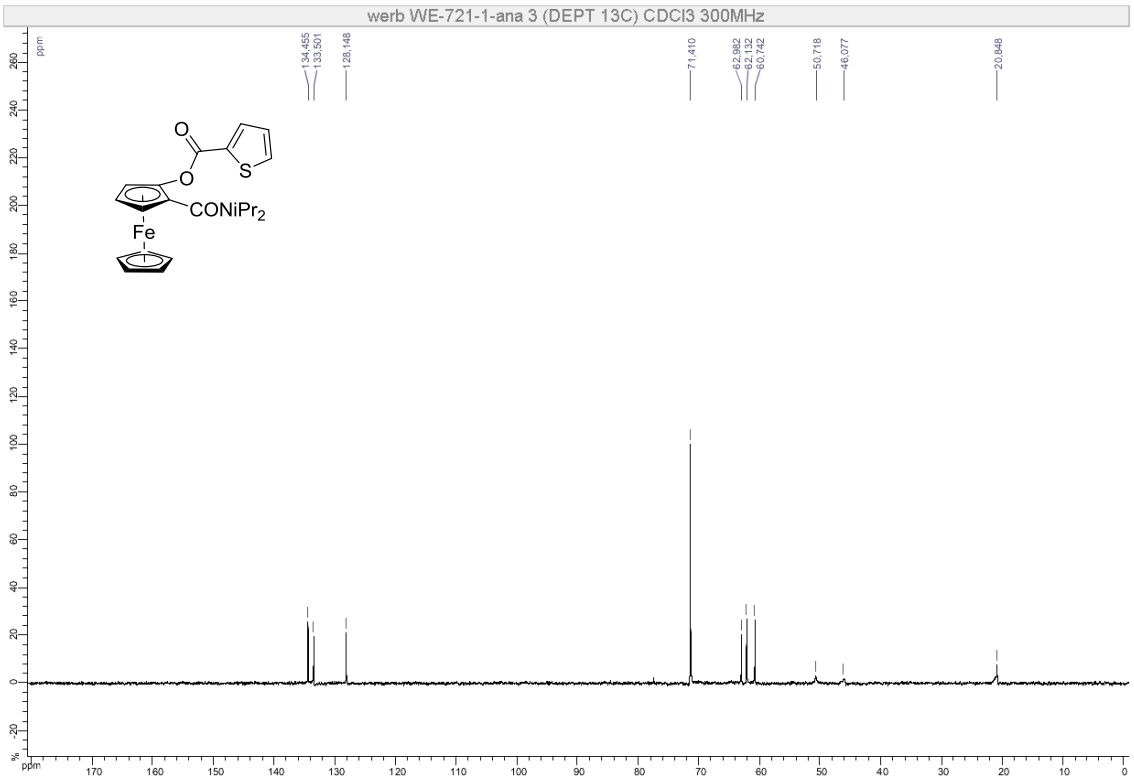

werb WE-723-1-ana 5 (HSQC 1H/13C) CDCl3 300MHz
werb WE-723-1-ana 1 (1D 1H)
O
O
Fe
CONiPr2
Fe
ppm
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5
werb WE-723-1-ana 2 (1D 13C)

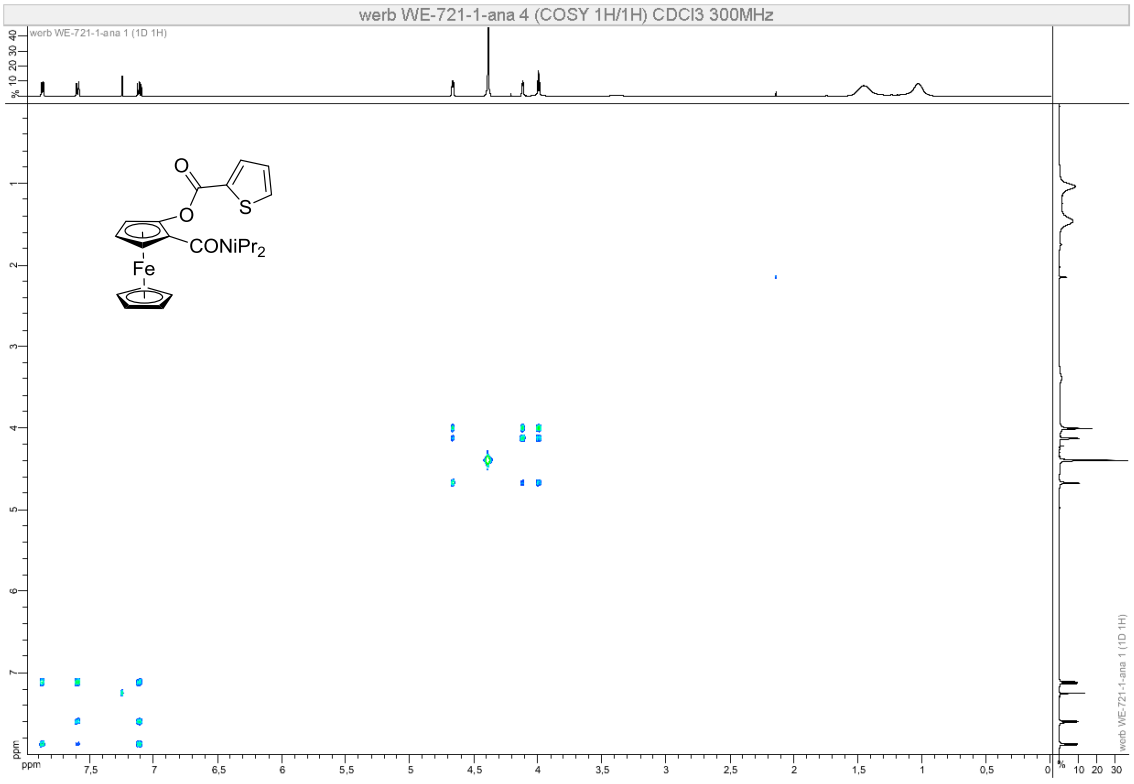

werb WE-723-1-ana 6 (HMBC 1H/13C) CDCl3 300MHz
werb WE-723-1-ana 1 (1D 1H)
O
O
Fe
CONiPr2
Fe
ppm
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5
werb WE-723-1-ana 2 (1D 13C)

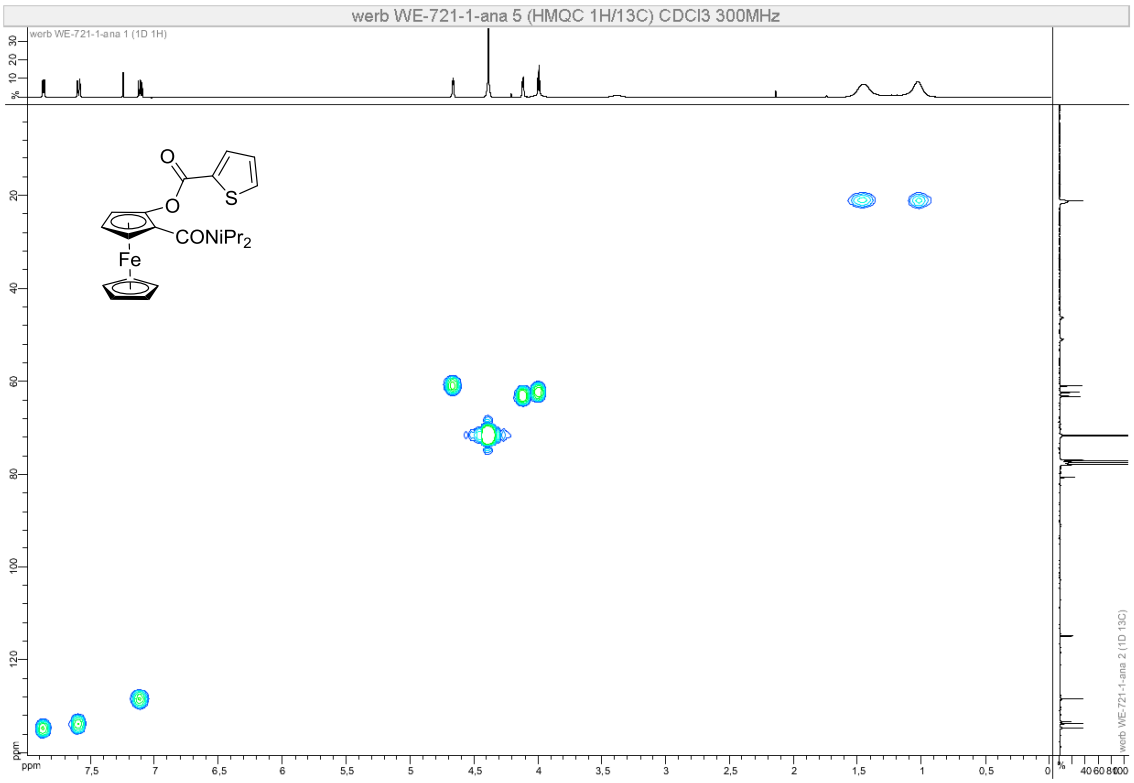
Compound (±)-17

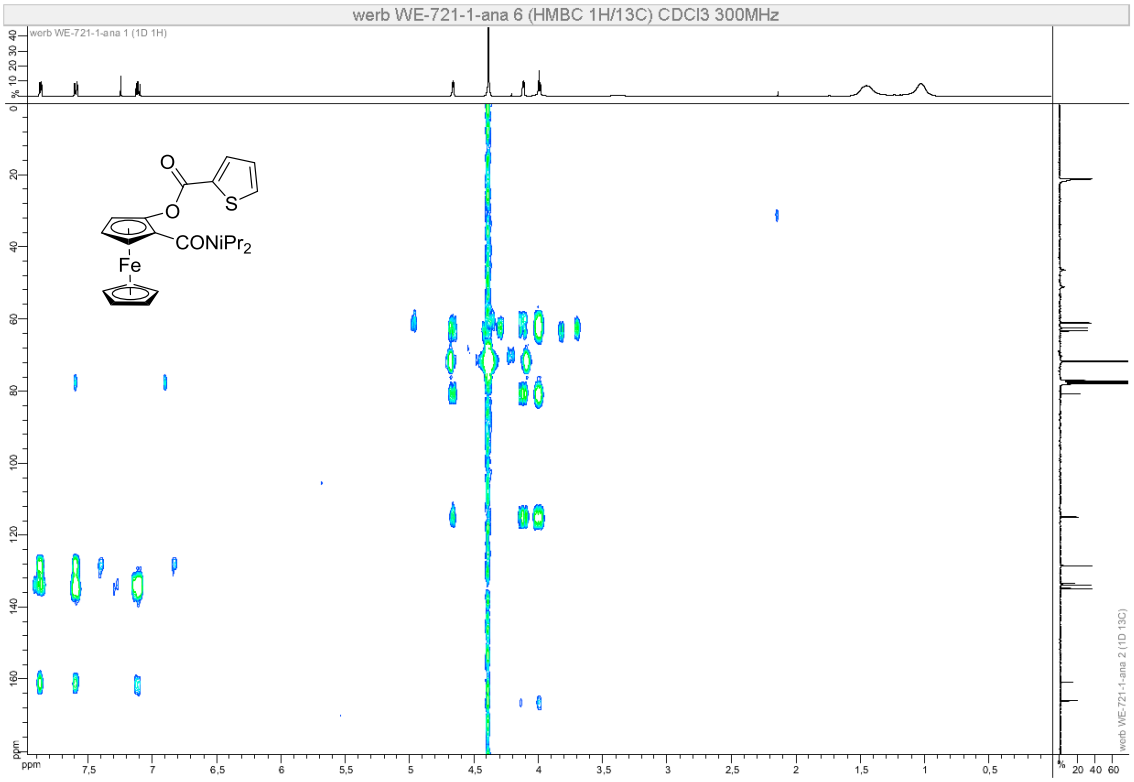

werb WE-721-1-ana 1 (1D 1H) CDCl3 300MHz
CONiPr2
Fe


werb WE-721-1-ana 2 (1D 13C) CDCl3 300MHz
CONiPr2
Fe

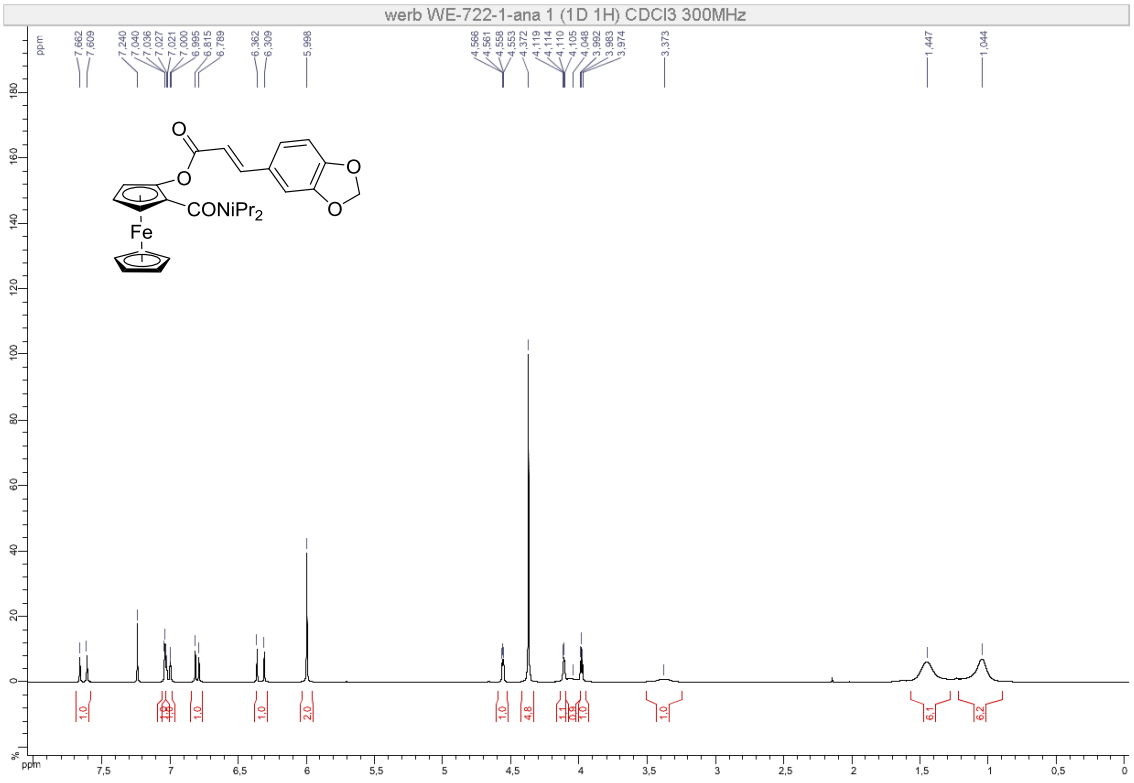

werb WE-721-1-ana 3 (DEPT 13C) CDCl3 300MHz
134,455
133,501
128,148
71,410
62,982
62,132
60,742
50,718
46,077
20,848
CONiPr2
Fe

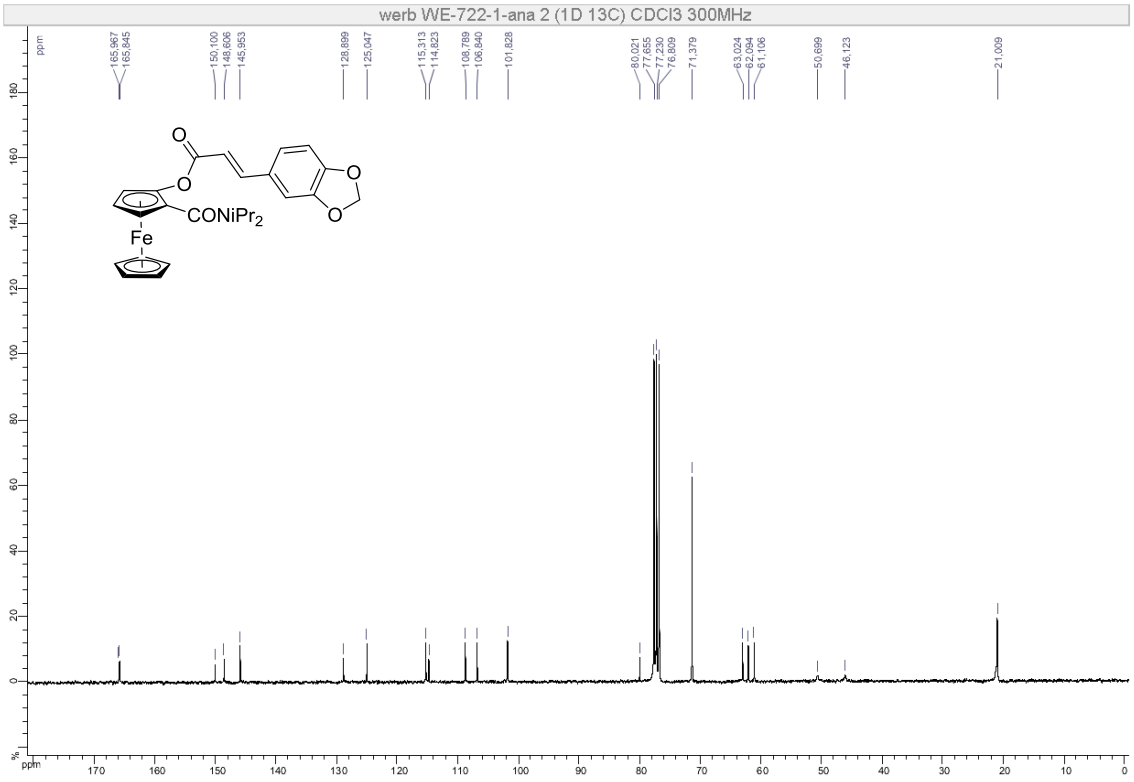

werb WE-721-1-ana 4 (COSY 1H/1H) CDCl3 300MHz
werb WE-721-1-ana 1 (1D 1H)
CONiPr2
Fe

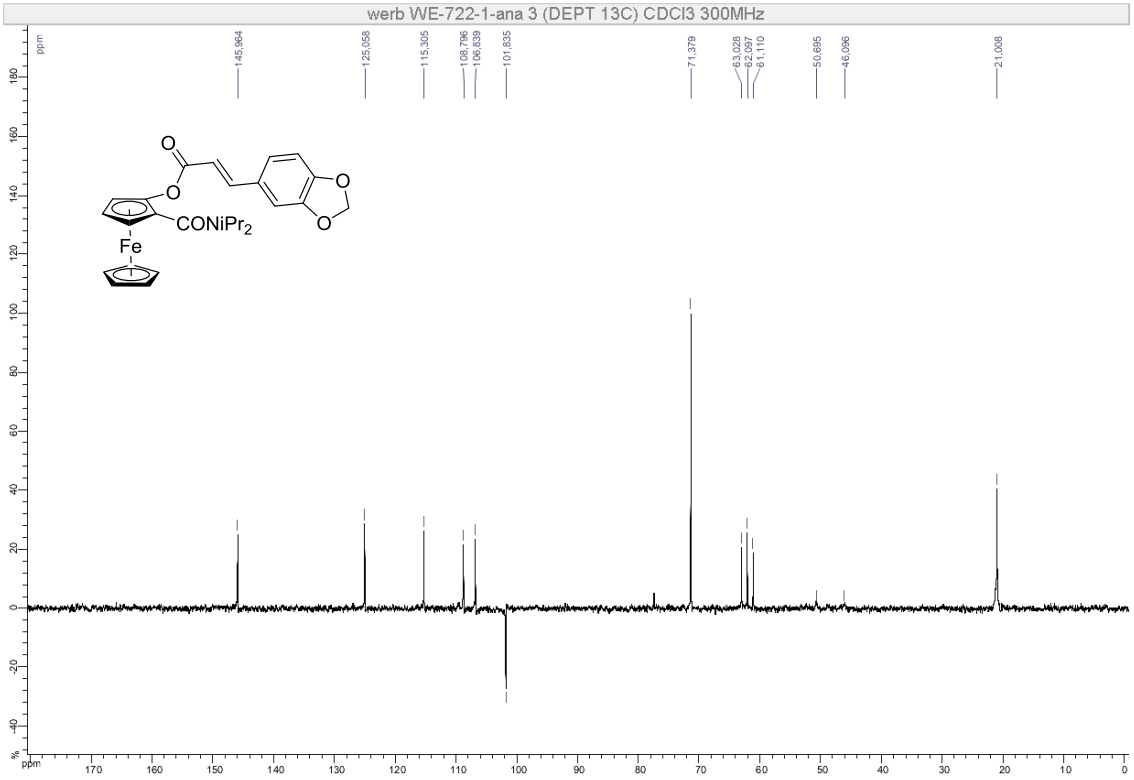

werb WE-721-1-ana 5 (HMQC 1H/13C) CDCl3 300MHz
werb WE-721-1-ana 1 (1D 1H)
O
S
O
CONiPr2
Fe
ppm
werb WE-721-1-ana 2 (1D 13C)

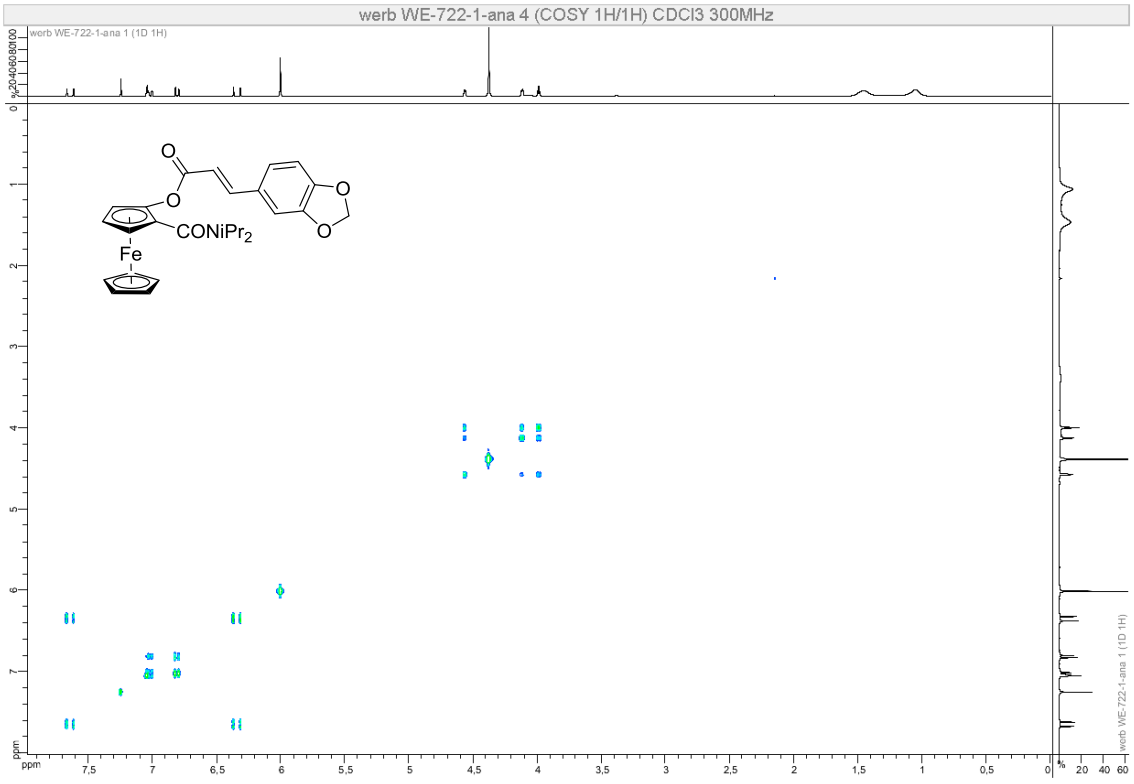

werb WE-721-1-ana 6 (HMBC 1H/13C) CDCl3 300MHz
werb WE-721-1-ana 1 (1D 1H)
O
S
O
CONiPr2
Fe
ppm
werb WE-721-1-ana 2 (1D 13C)

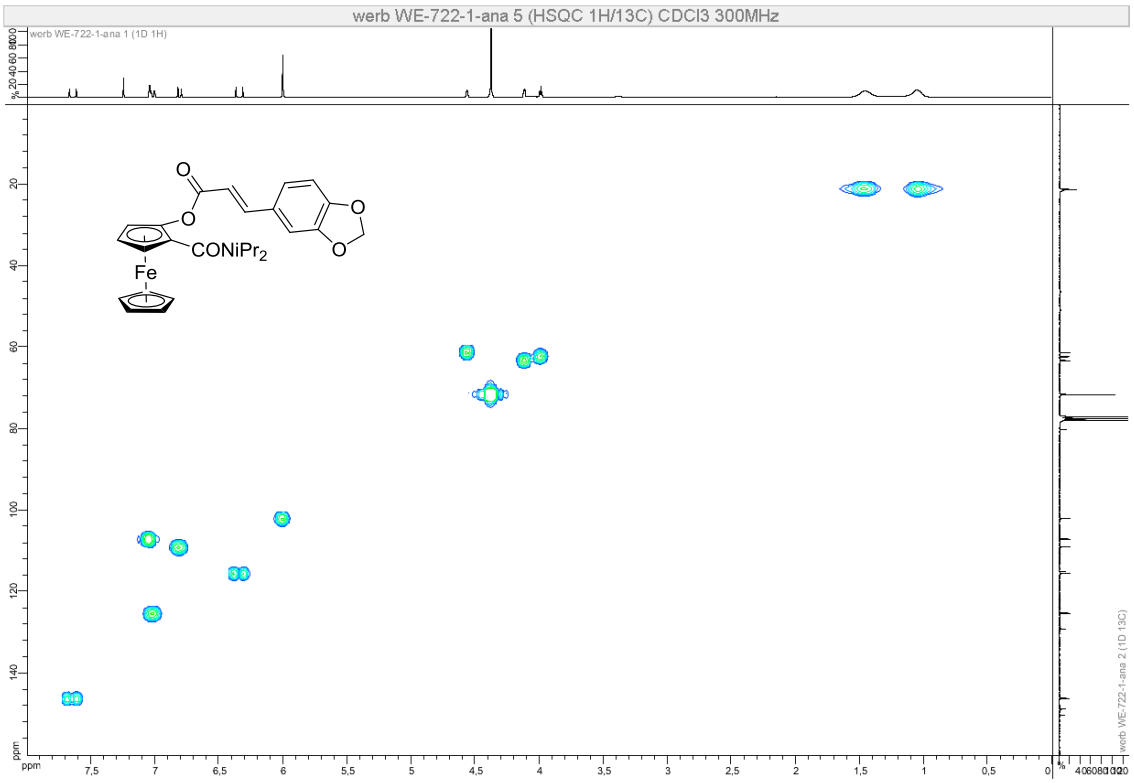
Compound (±)-18

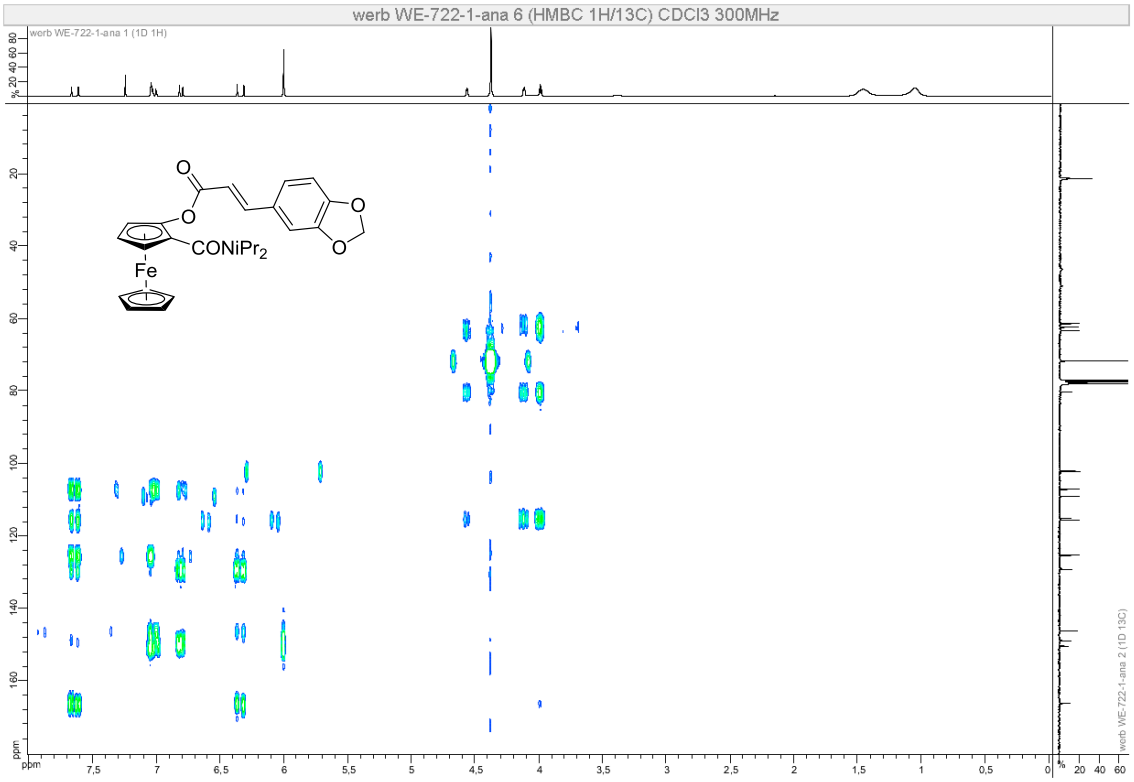

werb WE-722-1-ana 1 (1D 1H) CDCl3 300MHz
CONiPr2
Fe


werb WE-722-1-ana 2 (1D 13C) CDCl3 300MHz
CONiPr2
Fe

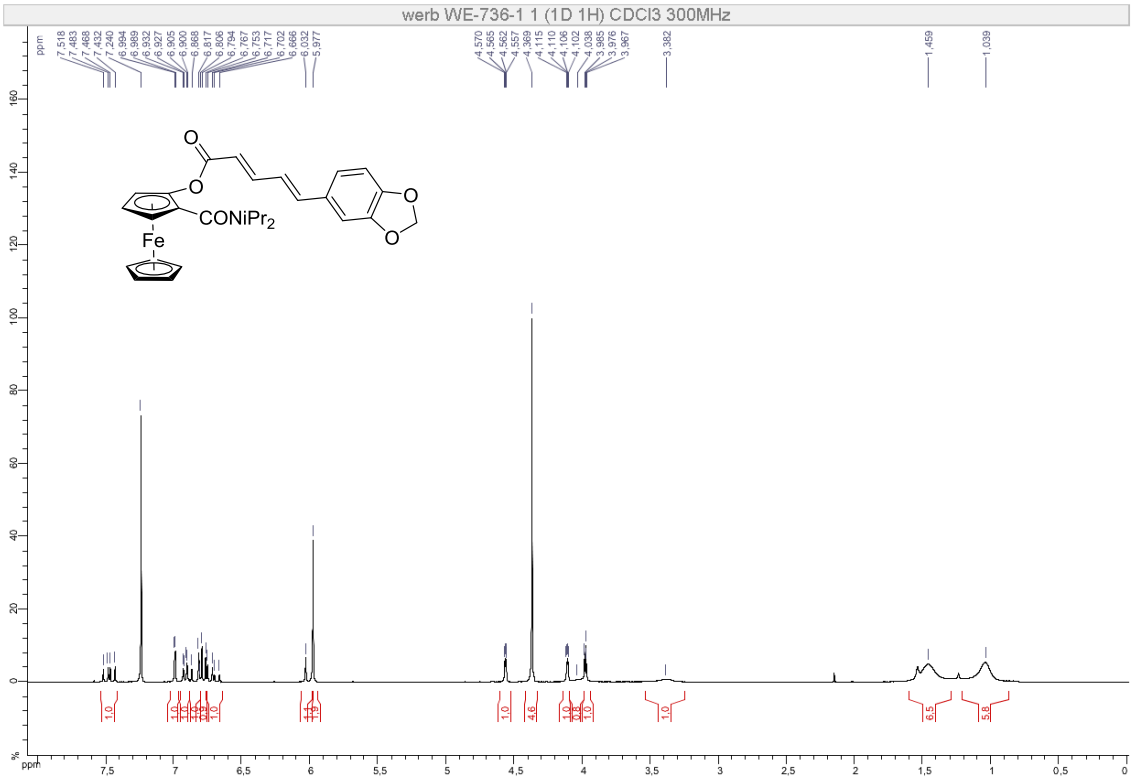

werb WE-722-1-ana 3 (DEPT 13C) CDCl3 300MHz
145,964
125,058
115,305
108,796
106,839
101,835
71,379
63,028
62,097
61,110
50,695
46,096
21,008
CONiPr2
Fe

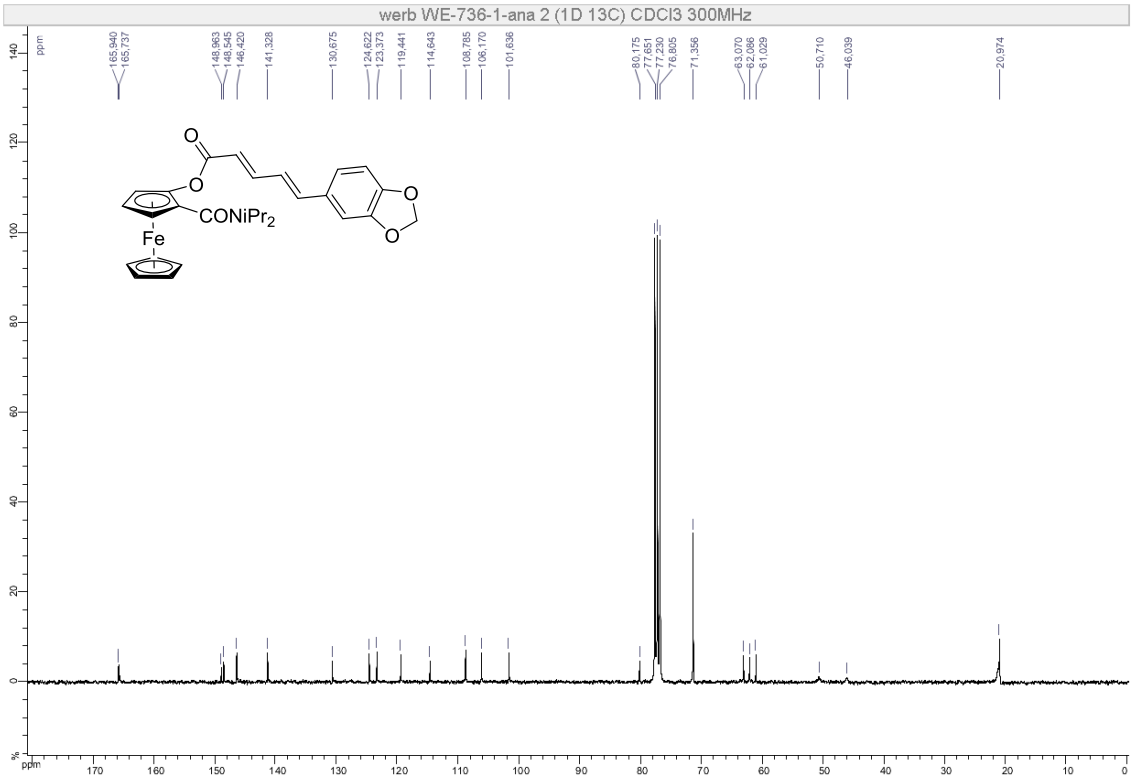

werb WE-722-1-ana 4 (COSY 1H/1H) CDCl3 300MHz
werb WE-722-1-ana 1 (1D 1H)
CONiPr2
Fe

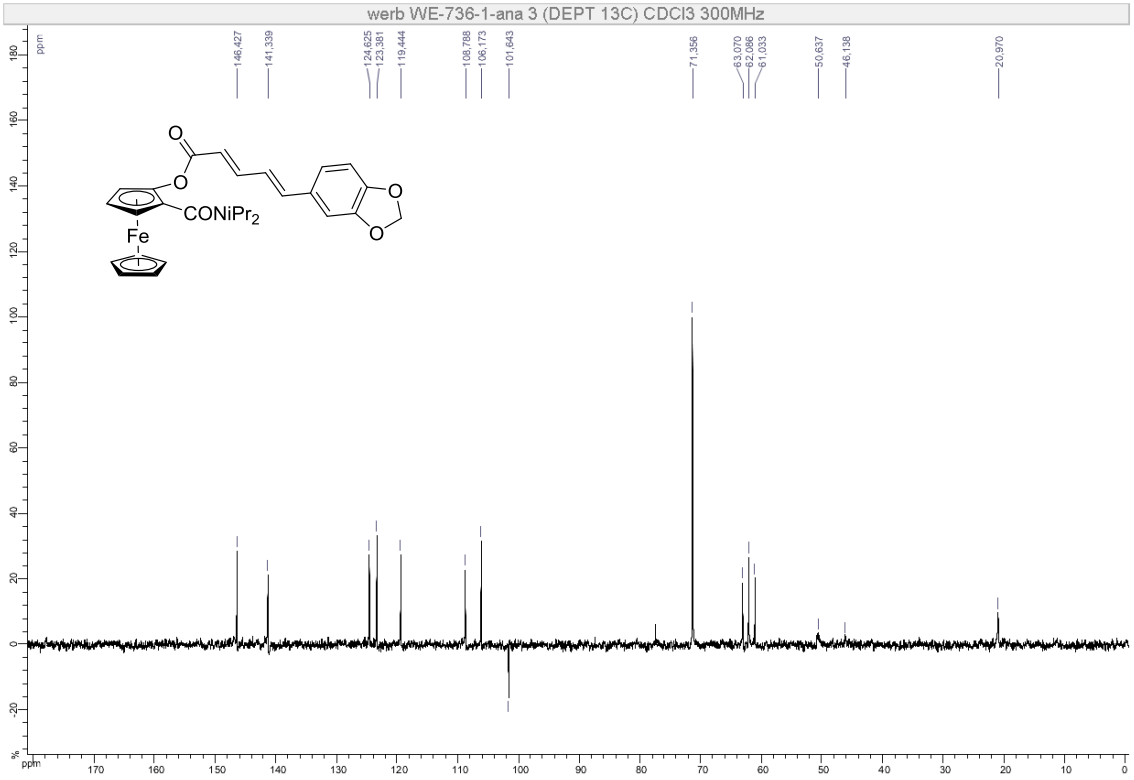

werb WE-722-1-ana 5 (HSQC 1H/13C) CDCl3 300MHz
werb WE-722-1-ana 1 (1D 1H)
werb WE-722-1-ana 2 (1D 13C)
ppm
O
O
O
O
CONiPr2
Fe

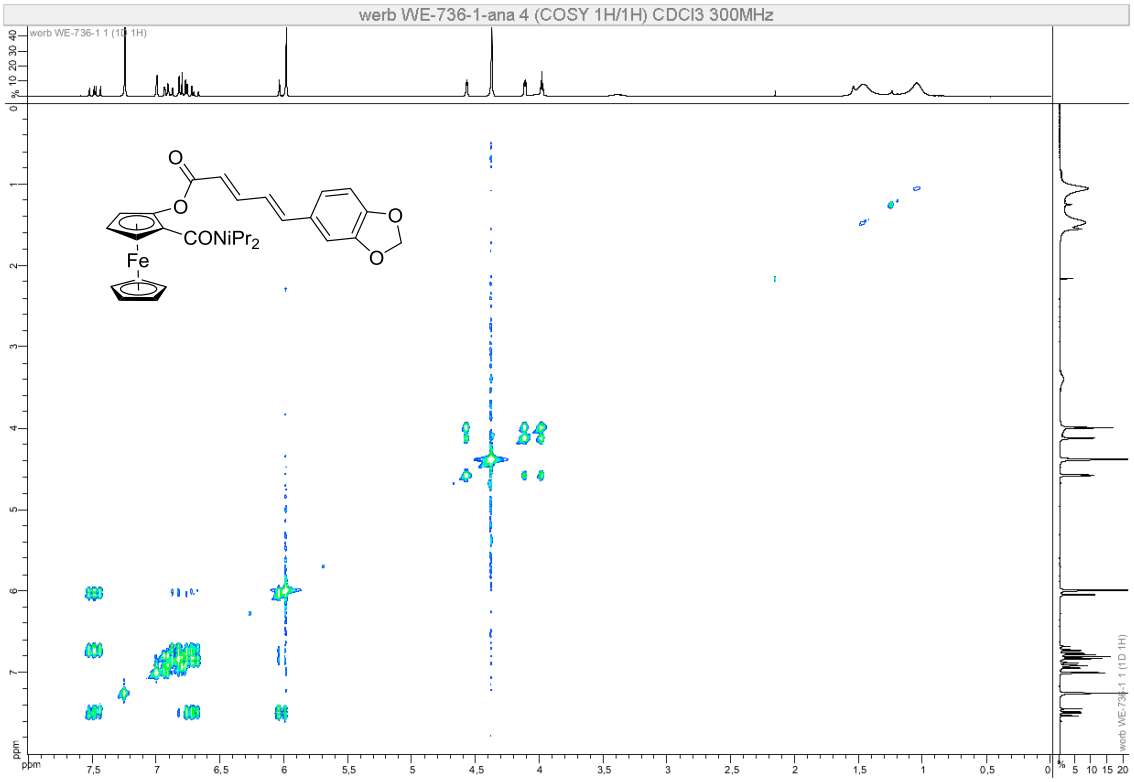

werb WE-722-1-ana 6 (HMBC 1H/13C) CDCl3 300MHz
werb WE-722-1-ana 1 (1D 1H)
werb WE-722-1-ana 2 (1D 13C)
ppm
O
O
O
O
CONiPr2
Fe

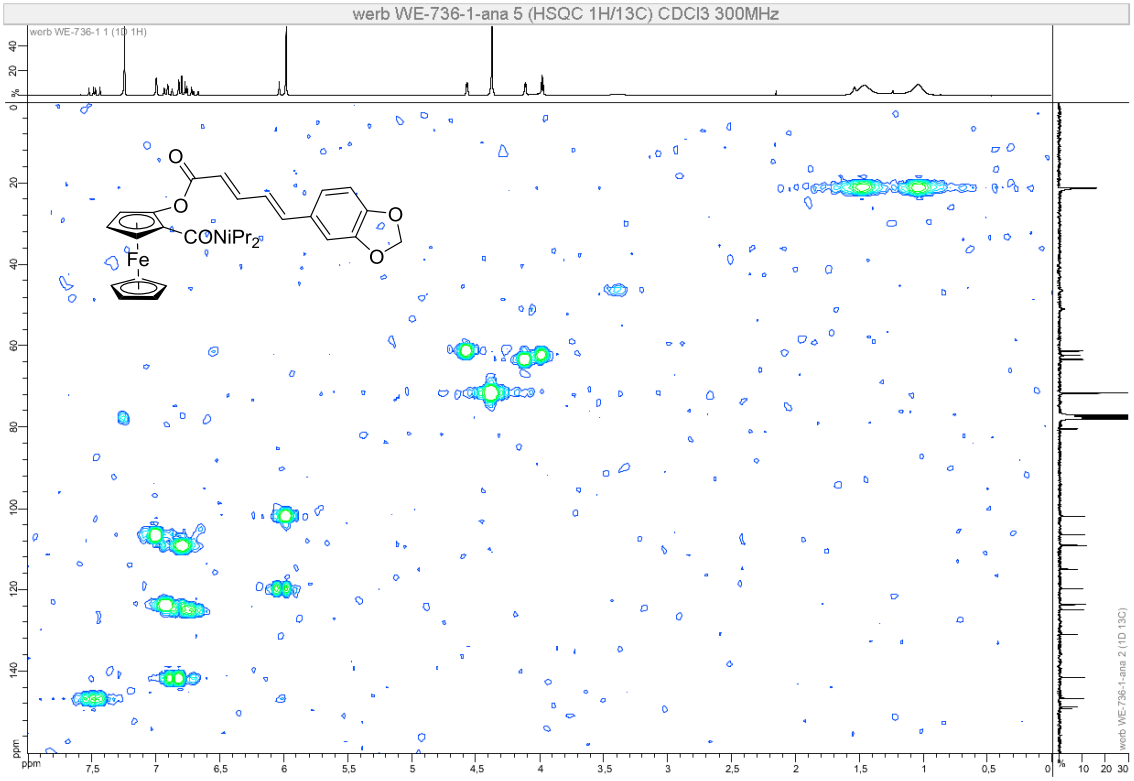
Compound (±)-19

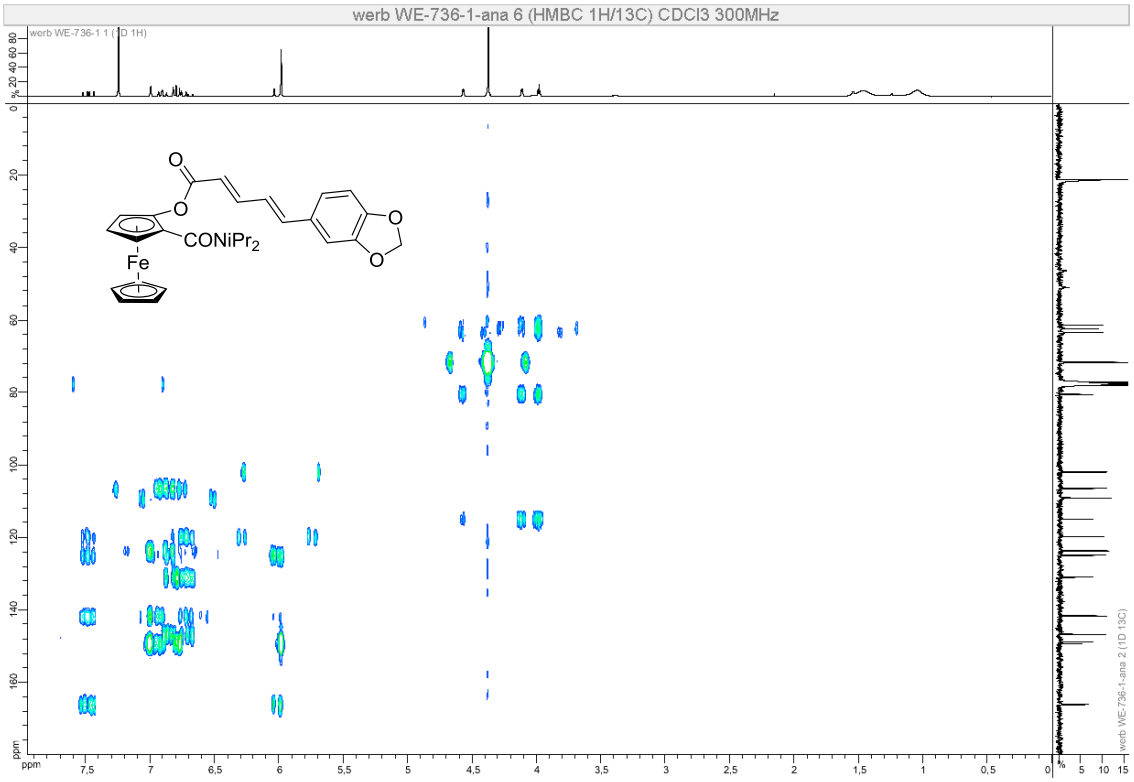

werb WE-736-1 1 (1D 1H) CDCl3 300MHz
CONiPr2
Fe


werb WE-736-1-ana 2 (1D 13C) CDCl3 300MHz
CONiPr2
Fe


werb WE-736-1-ana 3 (DEPT 13C) CDCl3 300MHz
ppm
146,427
141,339
124,625
123,381
119,444
108,788
106,173
101,643
71,356
63,070
62,086
61,033
50,637
46,138
20,970
O
O
CONiPr2
Fe
O
O
%
ppm


werb WE-736-1-ana 4 (COSY 1H/1H) CDCl3 300MHz
werb WE-736-1 1 (1D 1H)
O
O
CONiPr2
Fe
O
O
ppm
werb WE-736-1 1 (1D 1H)


werb WE-736-1-ana 5 (HSQC 1H/13C) CDCl3 300MHz
CONiPr2
Fe
O
O
O
O
ppm
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5
0


werb WE-736-1-ana 6 (HMBC 1H/13C) CDCl3 300MHz
CONiPr2
Fe
O
O
O
O
ppm
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5
0

Selected NOESY correlations observed by 2D NMR

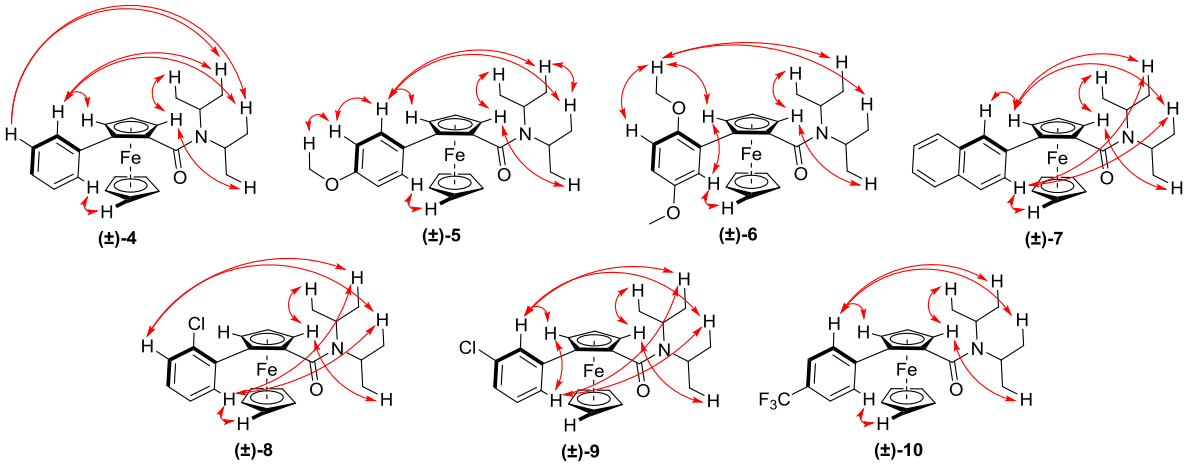
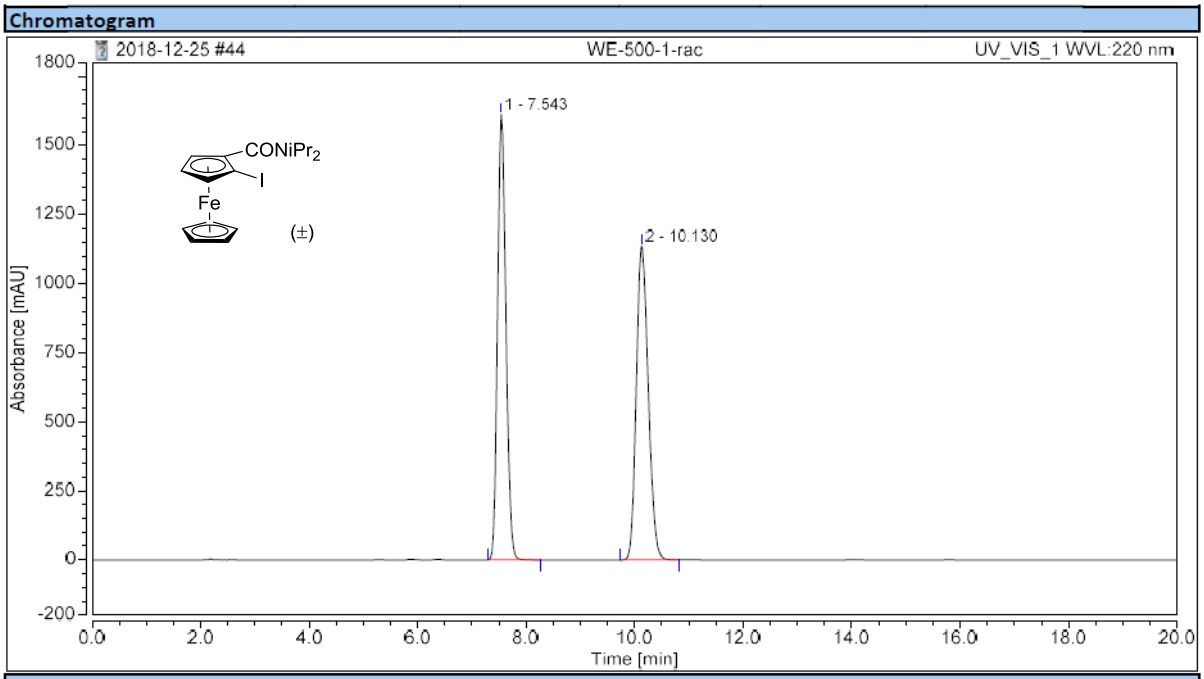


Figure 17.

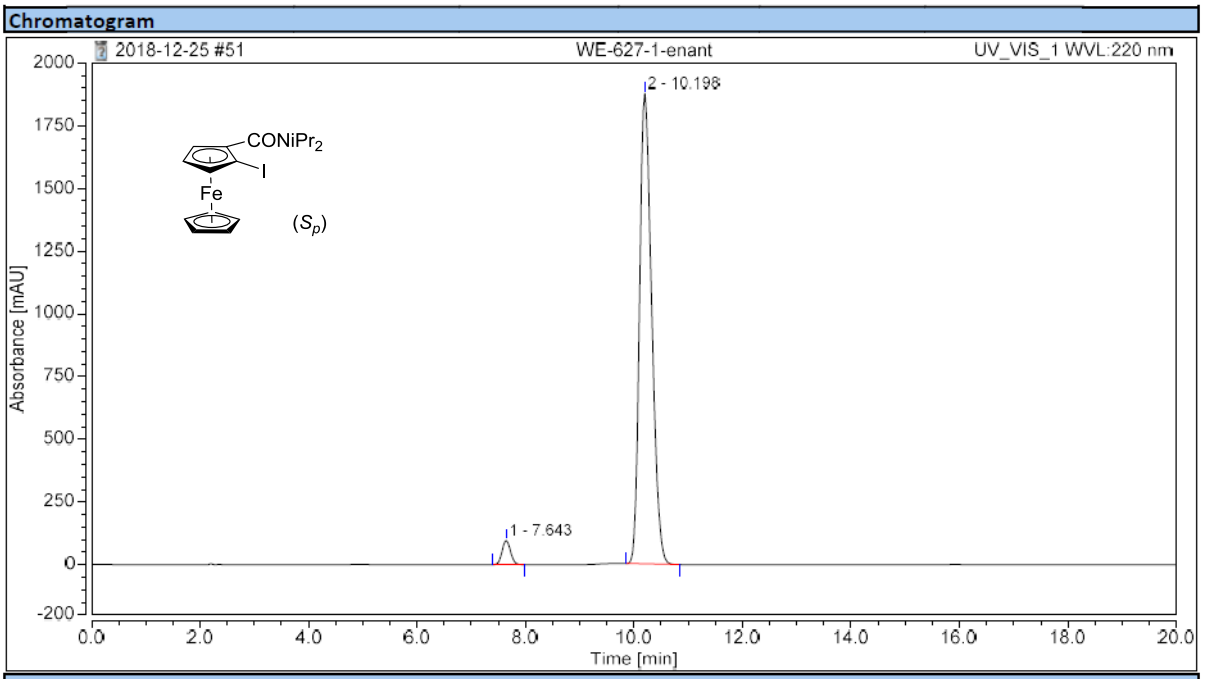
HPLC Chromatograms

Compounds (±)- and (S_p)-2



Integration Results

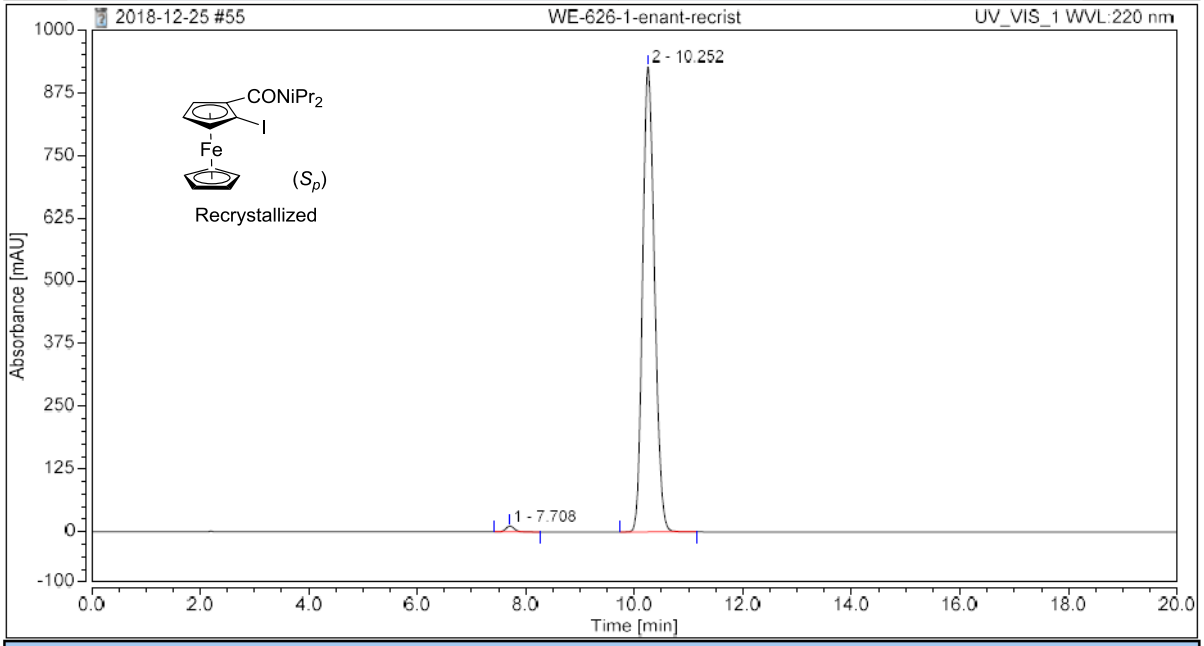
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		7.543	288.660	1612.105	49.99	58.66	n.a.
2		10.130	288.764	1135.984	50.01	41.34	n.a.
Total:			**577.424**	**2748.089**	**100.00**	**100.00**	



Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		7.643	17.482	97.481	3.48	4.94	n.a.
2		10.198	484.364	1875.323	96.52	95.06	n.a.
Total:			**501.846**	**1972.805**	**100.00**	**100.00**	

Chromatogram

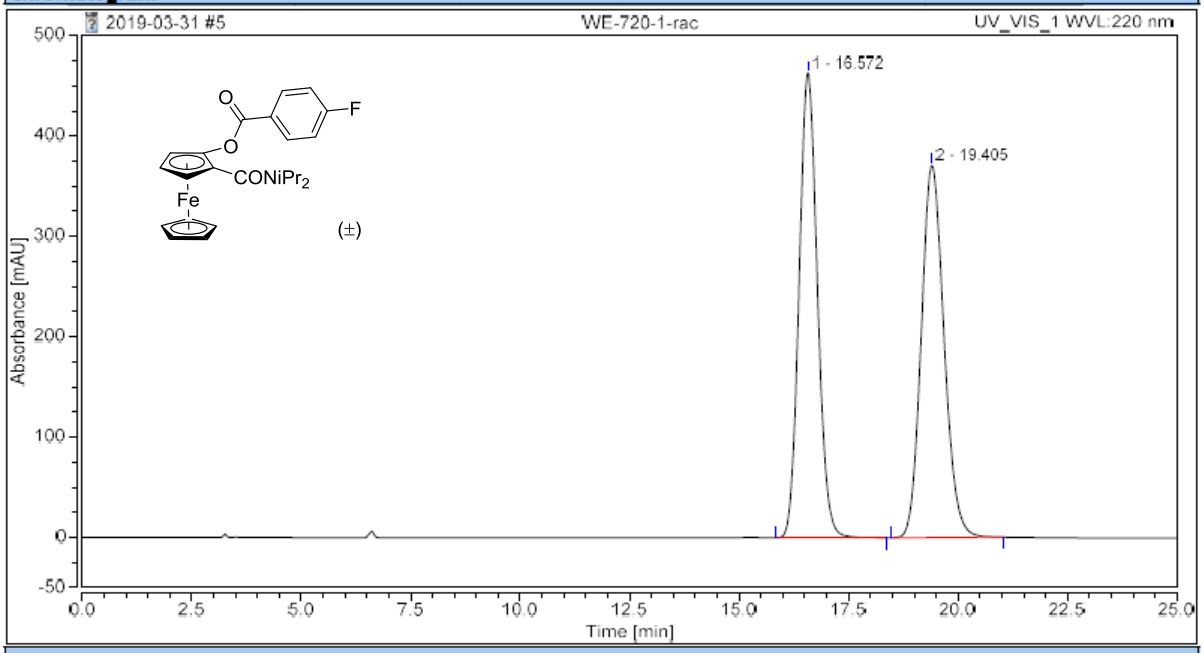


Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		7.708	2.090	11.604	0.88	1.24	n.a.
2		10.252	235.014	927.033	99.12	98.76	n.a.
Total:			**237.104**	**938.637**	**100.00**	**100.00**	

Compounds (±)- and (S_p)-11

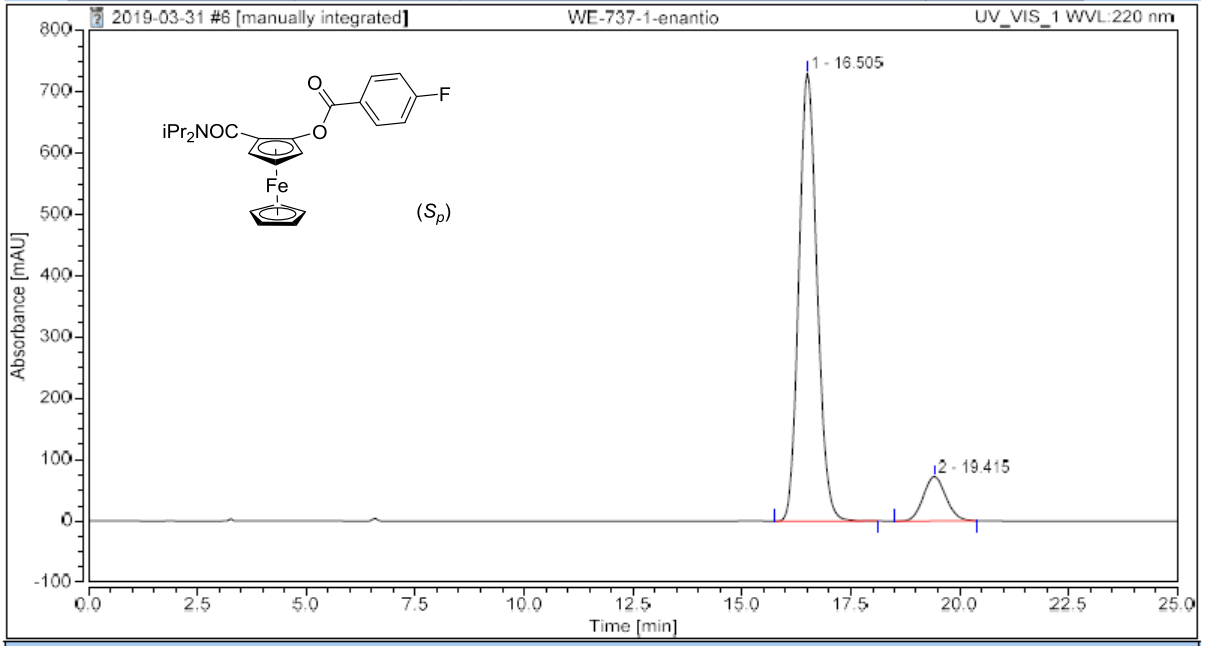
Chromatogram



Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		16.572	224.280	463.395	50.13	55.53	n.a.
2		19.405	223.120	371.025	49.87	44.47	n.a.
Total:			**447.400**	**834.420**	**100.00**	**100.00**	

Chromatogram



Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		16.505	354.328	730.746	89.06	90.99	n.a.
2		19.415	43.507	72.322	10.94	9.01	n.a.
Total:			**397.835**	**803.068**	**100.00**	**100.00**	

References

1. Sheldrick, G. *Acta Crystallogr. Sect. A* **2015**, *71*, 3-8.
2. Sheldrick, G. *Acta Crystallogr. C* **2015**, *71*, 3-8.