

Supporting Information

Design and Synthesis of Ferrocenyl 1,4-Dihydropyridines and Their Evaluation as Kinesin-5 Inhibitors

Karolina Kowalczyk¹, Andrzej Błaż², Krzysztof Krawczyk², Błażej Rychlik², Damian Plażuk¹

¹ *Laboratory of Molecular Spectroscopy, Department of Organic Chemistry, Faculty of Chemistry, University of Lodz, ul. Tamka 12, 91-403 Łódź, Poland*

² *Cytometry Lab, Department of Oncobiology and Epigenetics, Faculty of Biology and Environmental Protection, University of Lodz, ul. Pomorska 141/143, 90-236 Łódź, Poland*

Table of Contents

Additional figures S1 and S2

¹H and ¹³C{¹H} NMR spectra for compounds **5**, **9a**, **10a-c**, **13e**, **14b-j**, **15a-j**, **16a-c**

HPLC-MS analysis for compounds **5**, **13e**, **14b-j**, **15a-j**, **16a-c**

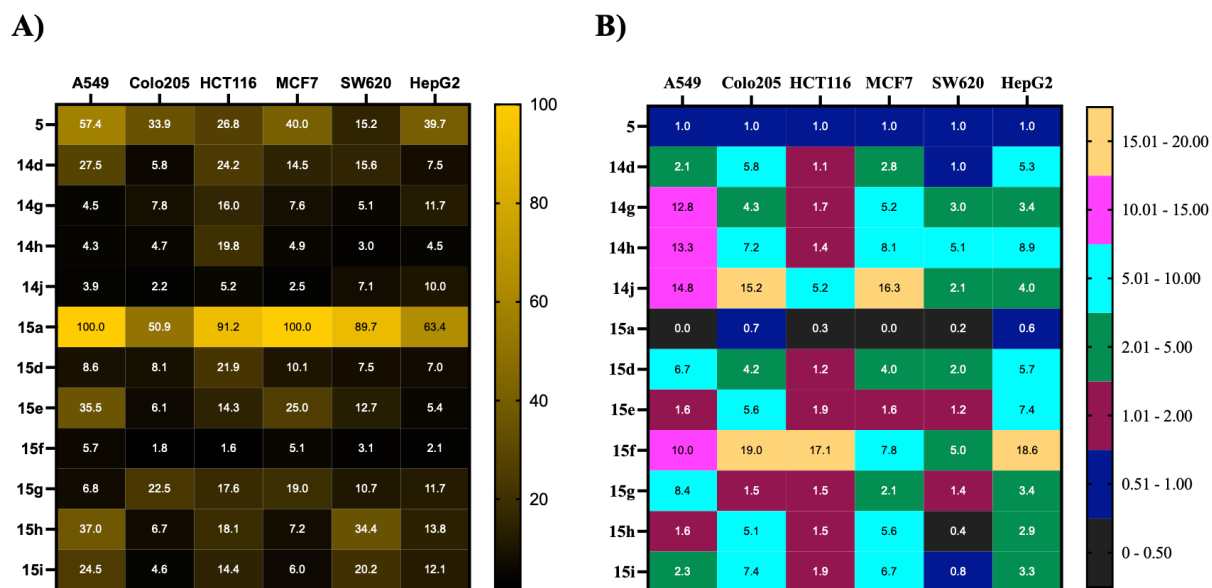


Figure S1. Antiproliferative activity **A)** Cytotoxic activity (IC_{50} values), **B)** The Activity Quotients (AQ) of studied compounds in comparison to cytotoxic activity of **5** were calculated as $AQ = IC_{50}(5)/IC_{50}(\text{compound})$

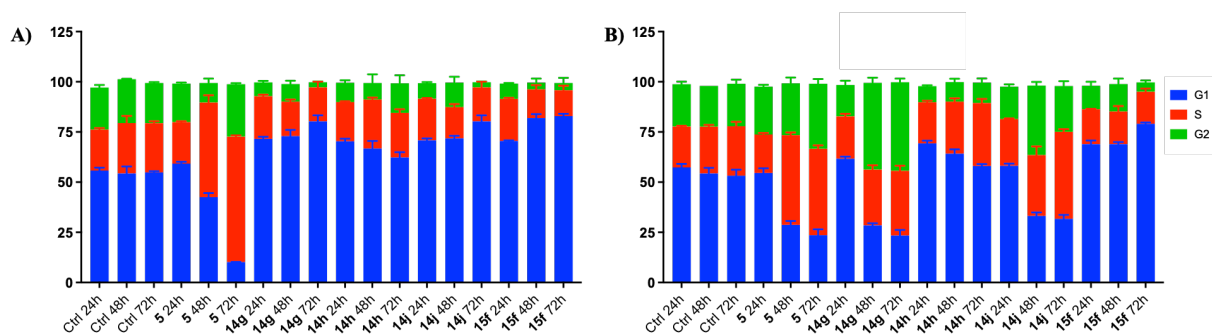


Figure S2. Cell cycle phase distribution for A549 (**A**) and SW620 (**B**) cells exposed for 24, 48 and 72h to **5** and its ferrocenyl analogues at concentration equal to the IC_{75} values for **5**. Data are presented as mean \pm SD, $n = 3$

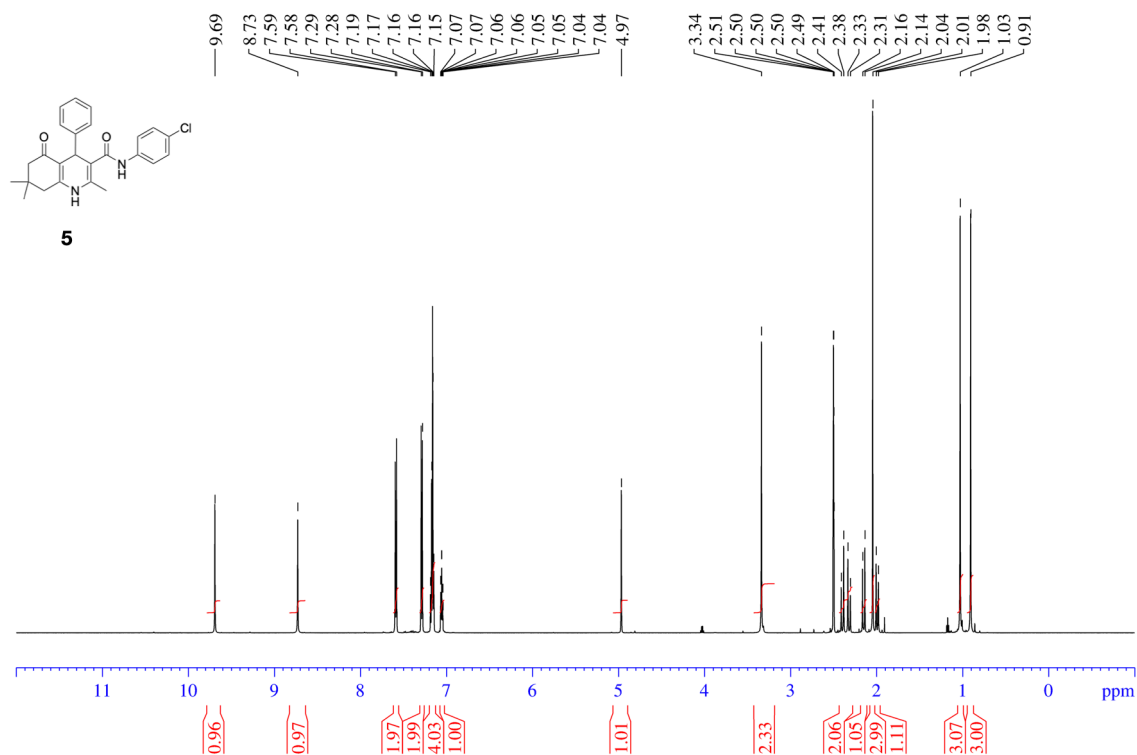


Figure S3. ¹H NMR spectrum of **5** in DMSO-d₆

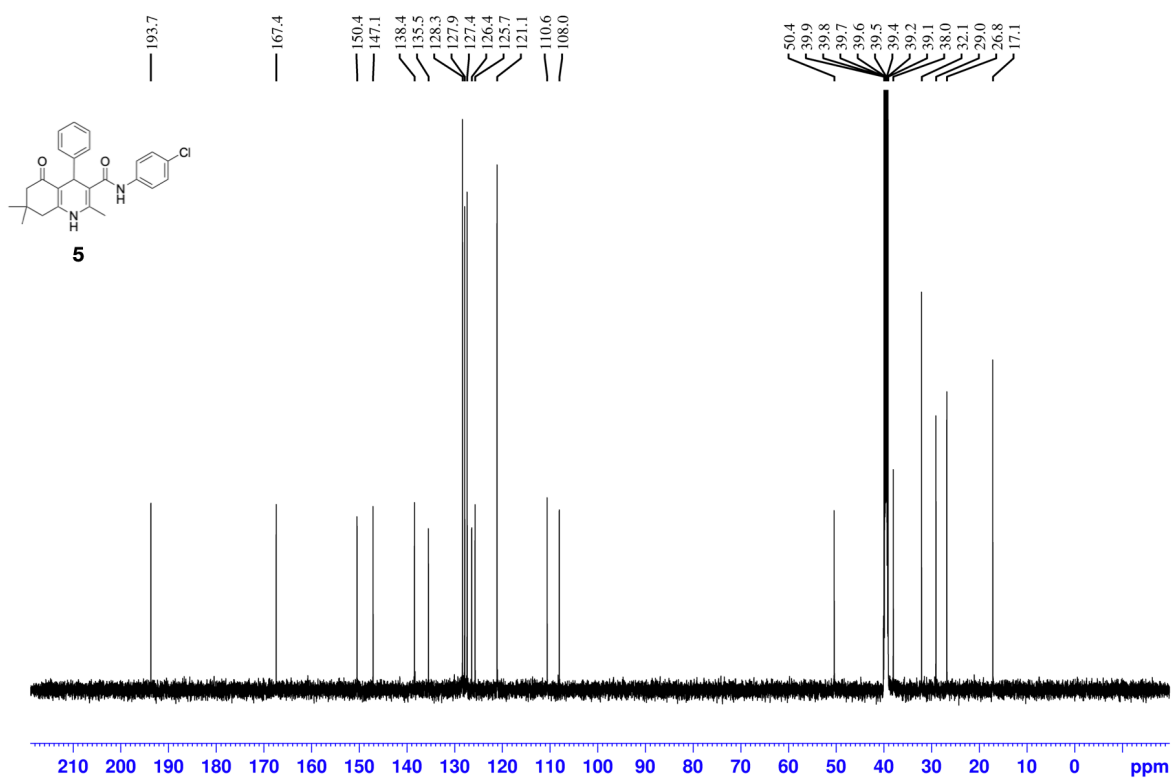


Figure S4. ¹³C{¹H} NMR spectrum of **5** in DMSO-d₆

Sample Type : Unknown
 Sample Name : KKG-90012
 Sample ID : KKG-90012
 Vial# : 84
 Injection Volume : 1

Method

<<LC Time Program>>

Time	Module	Command	Valu
4.00	Pumps	B.Conc	90
7.00	Pumps	B.Conc	90
7.00	Pumps	B.Conc	90
13.00	Controller	Stop	50

<<Mobile Phase Name>>

Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH

<<Data Acquisition>>

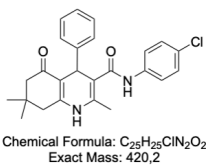
LC Stop Time : 13.00 min

<<Pump>>

Total Flow : 0.3000 mL/min

Peak Table

Peak#	Ret. Time	Area%
1	7.208	0.925
2	7.513	98.160
3	8.138	0.915
Total		100.000



5

Figure S5. HPLC-MS analysis of 5

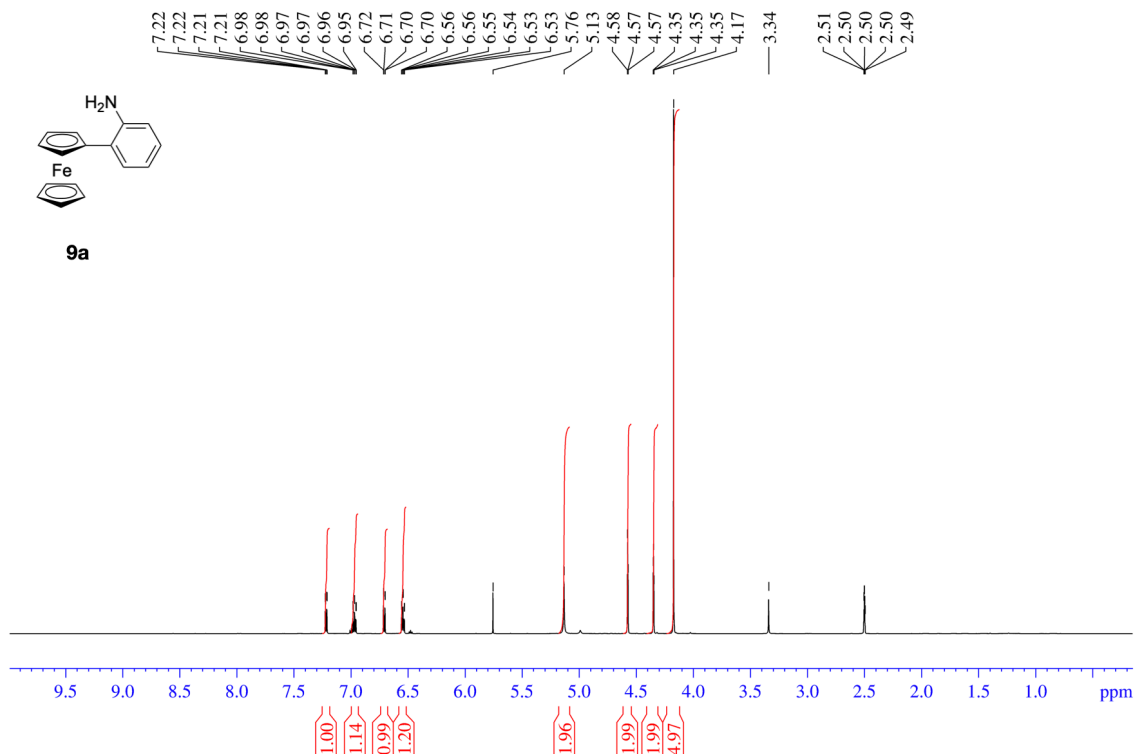
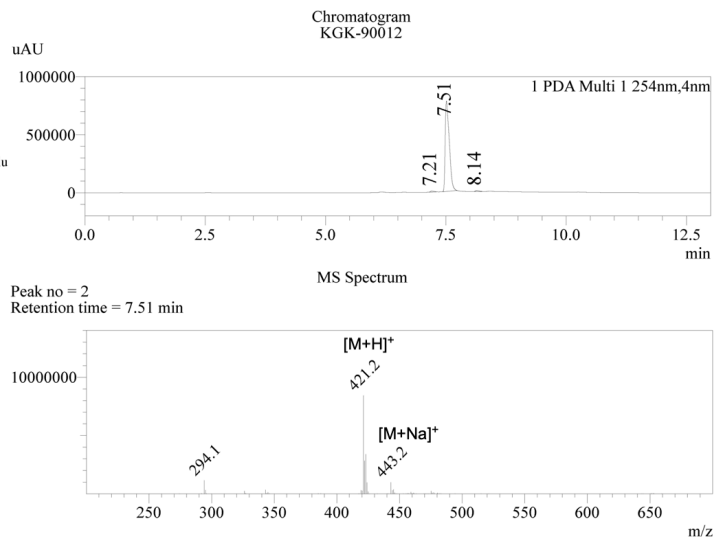


Figure S6. ¹H NMR spectrum of 9a in DMSO-d₆

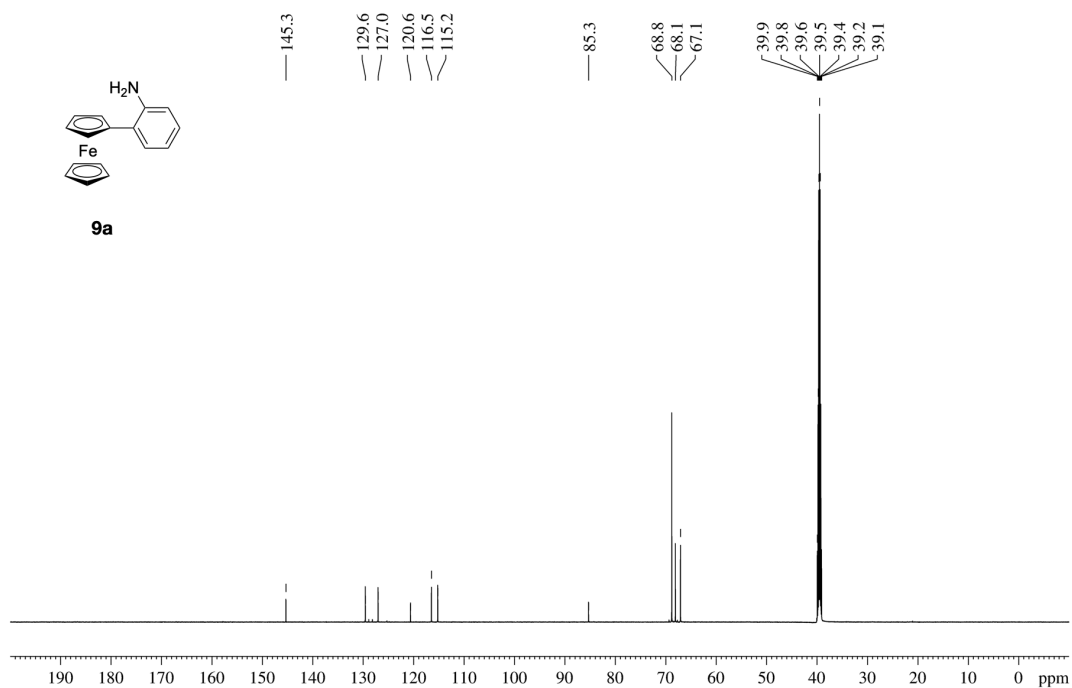


Figure S7. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **9a** in DMSO-d_6

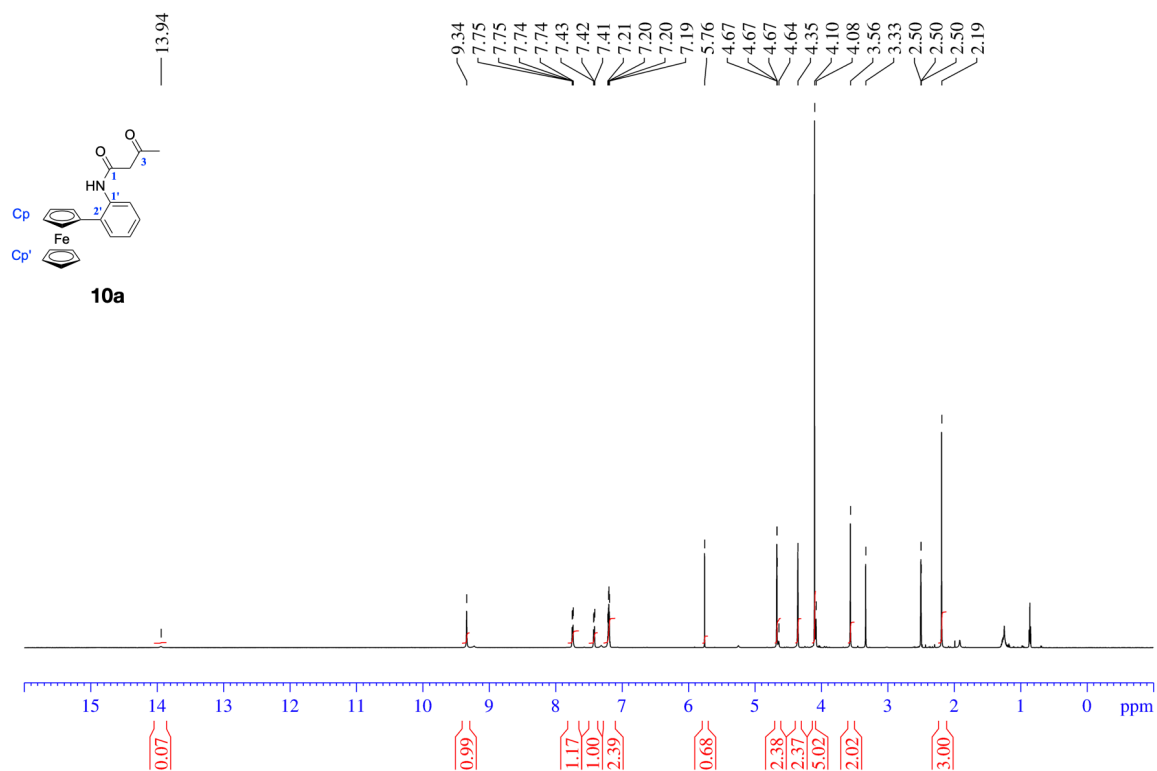


Figure S8. ^1H NMR spectrum of **10a** in DMSO-d_6

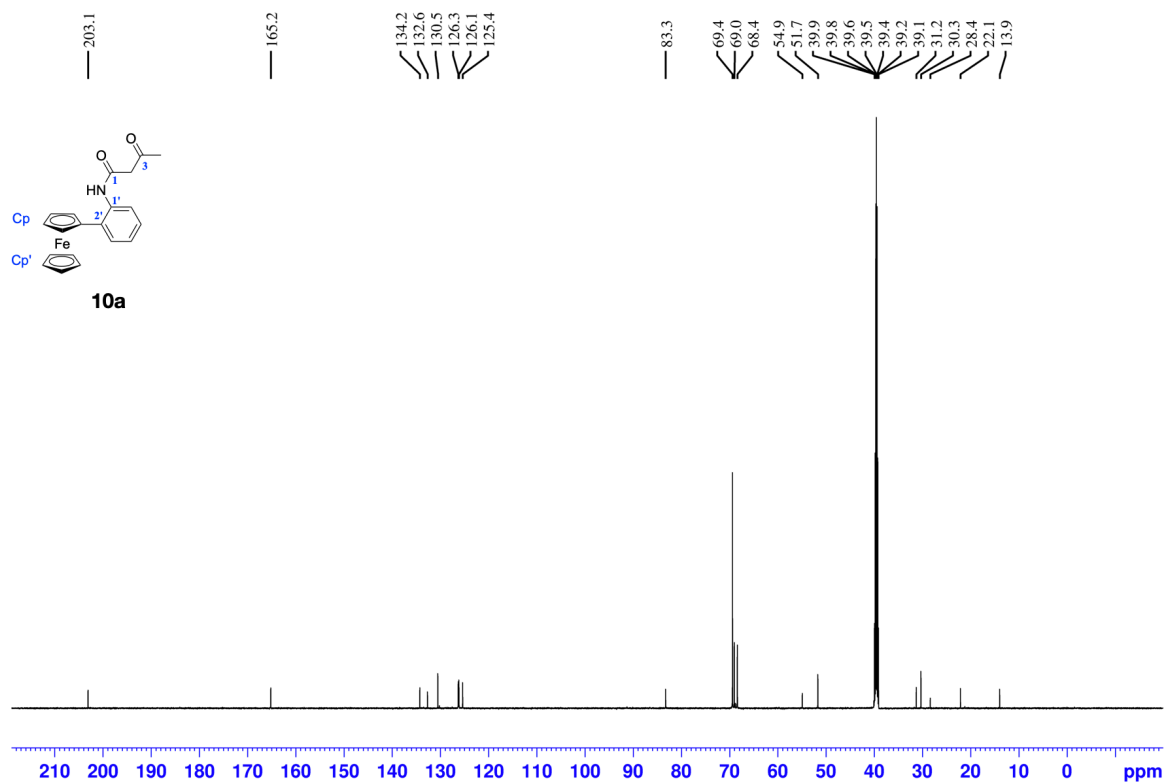


Figure S9. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10a** in DMSO-d_6

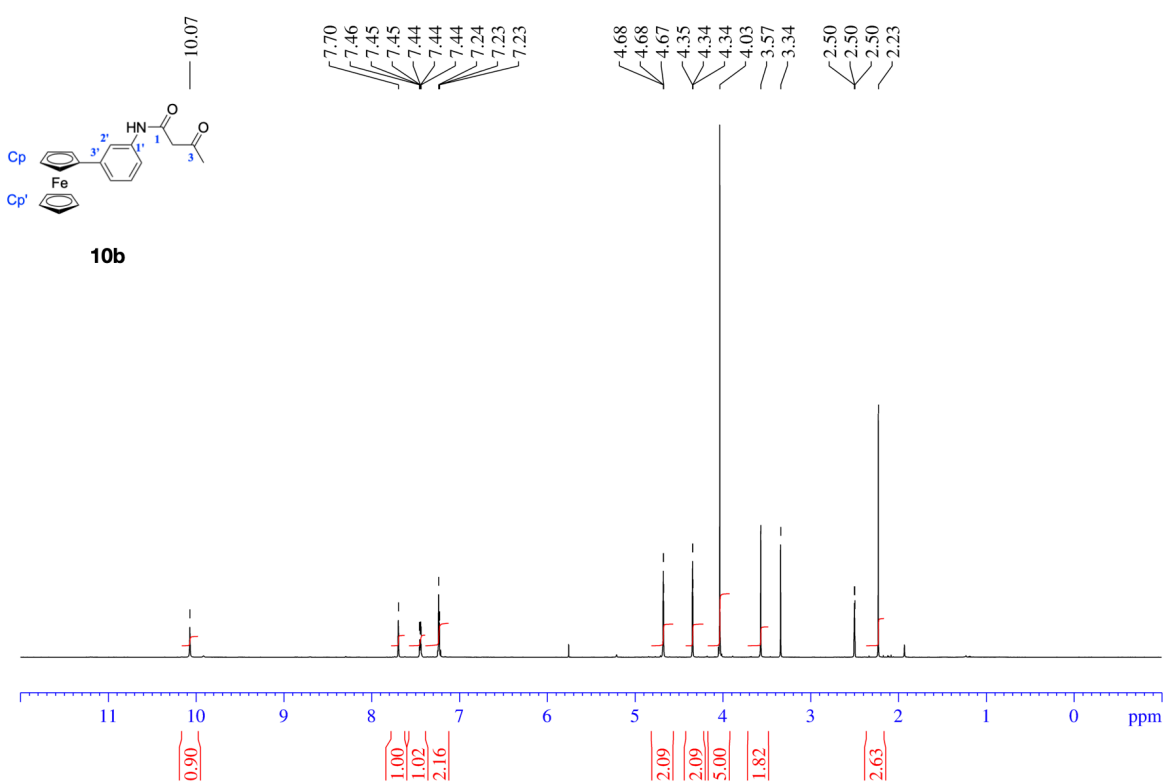


Figure S10. ^1H NMR spectrum of **10b** in DMSO-d_6

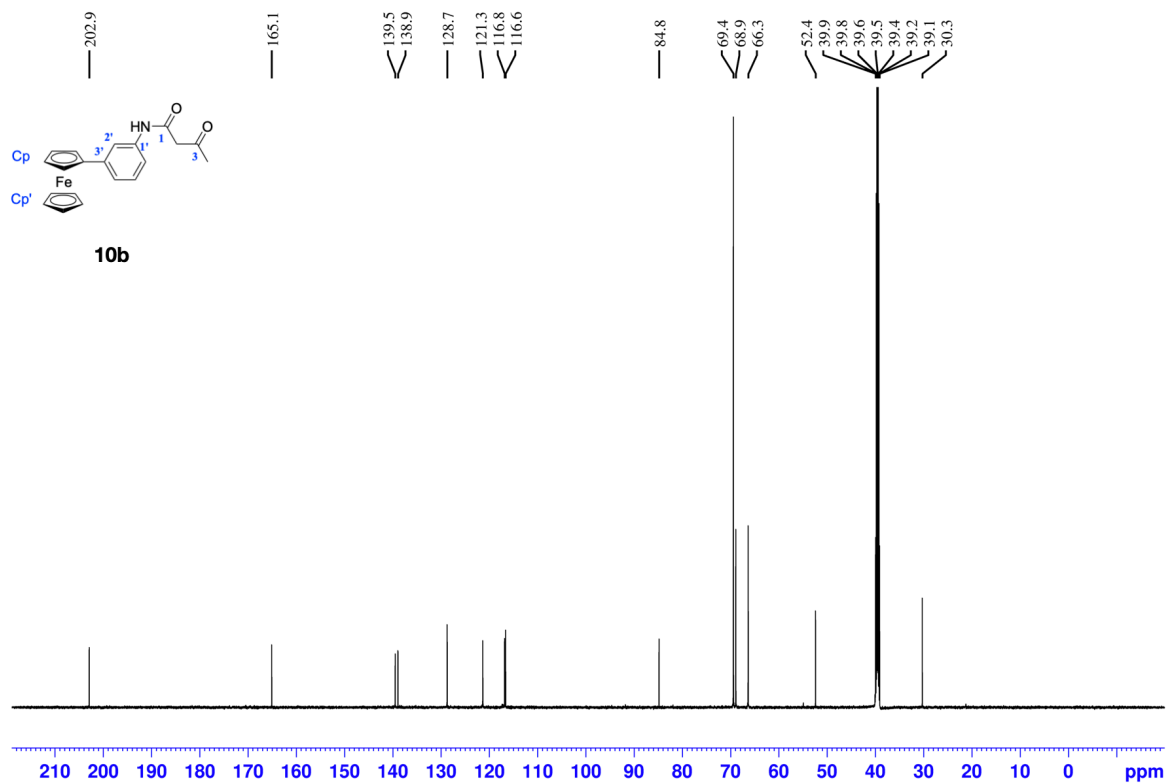


Figure S11. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10b** in DMSO-d_6

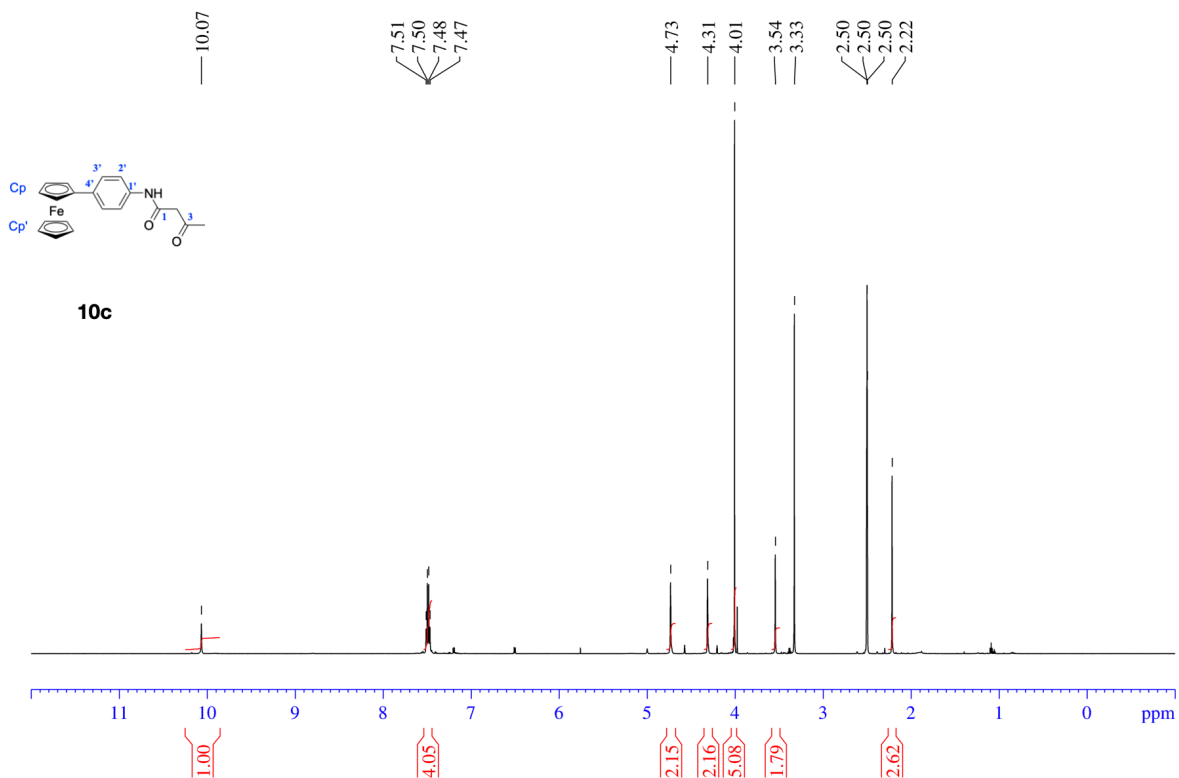


Figure S12. ^1H NMR spectrum of **10c** in DMSO-d_6

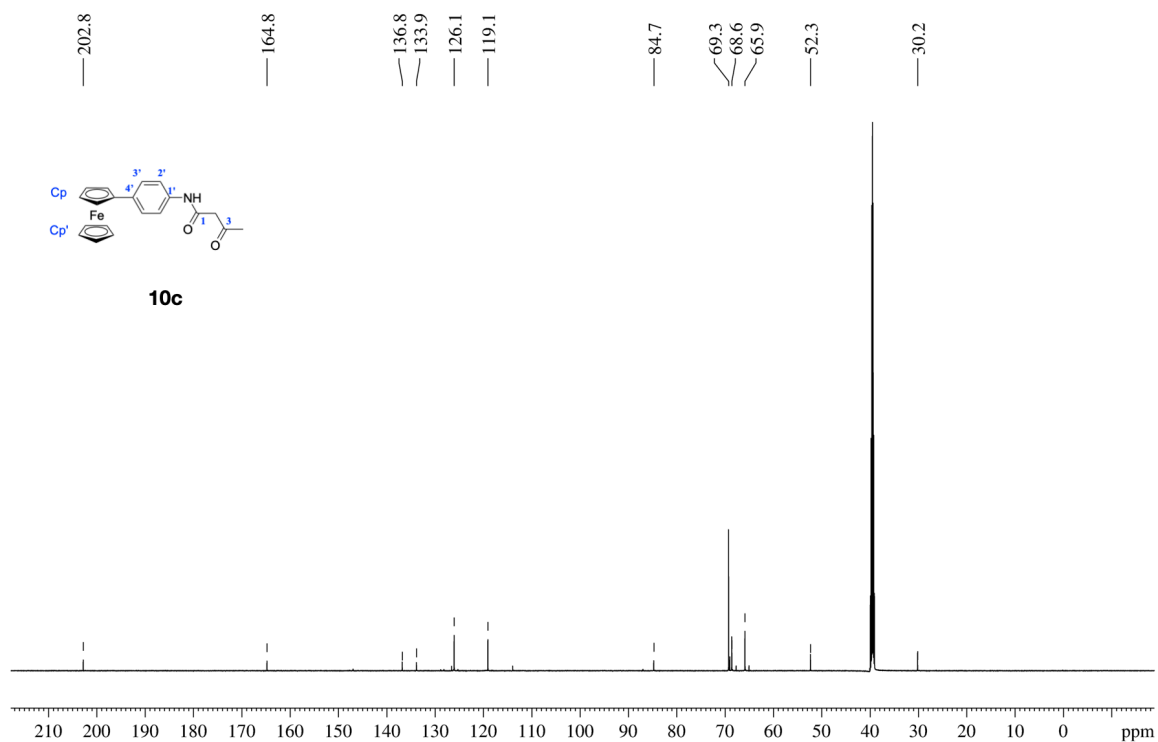


Figure S13. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10c** in DMSO-d_6

DMSO - 300K

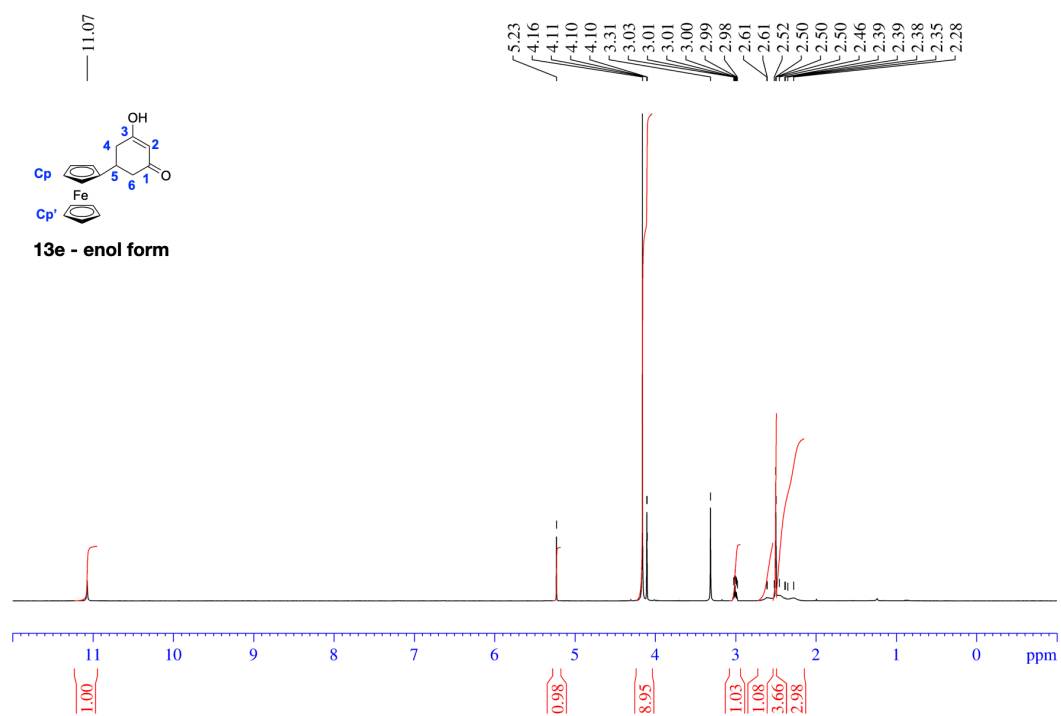


Figure S14. ^1H NMR spectrum of **13e** in DMSO-d_6

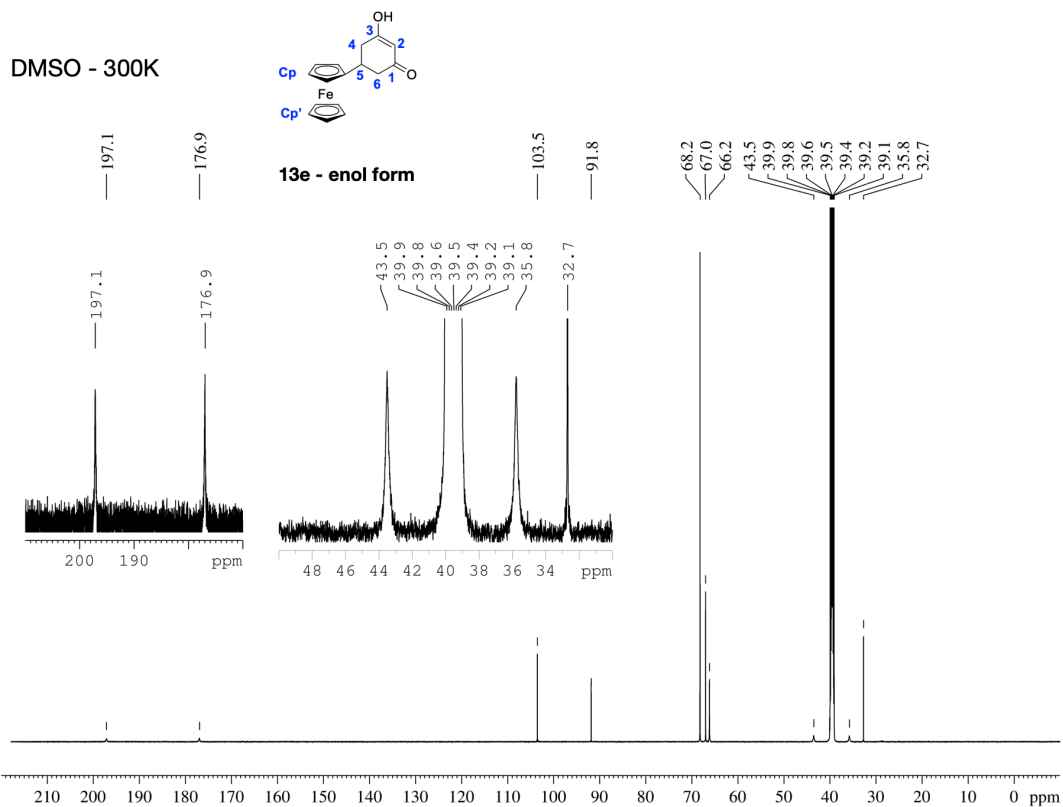


Figure S15. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **13e** in DMSO-d_6

CD_2Cl_2 - 300K

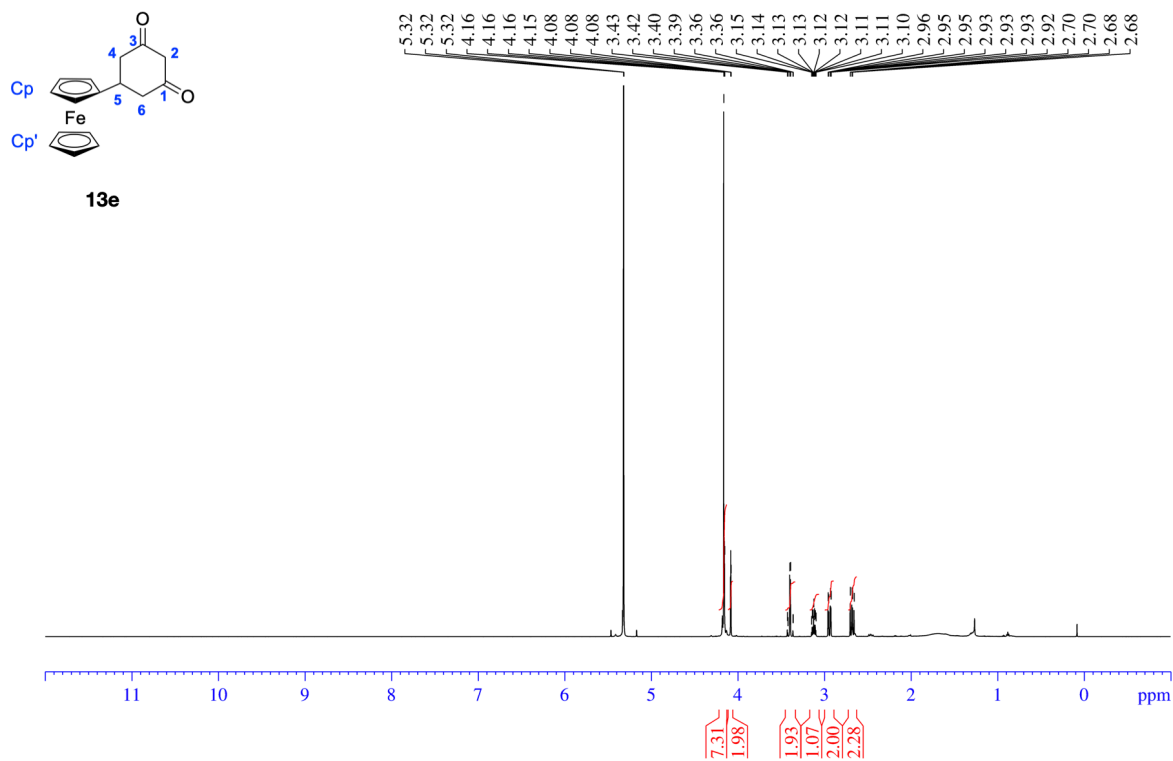


Figure S16. ^1H NMR spectrum of **13e** in DMSO-d_6

CD₂Cl₂ - 300K

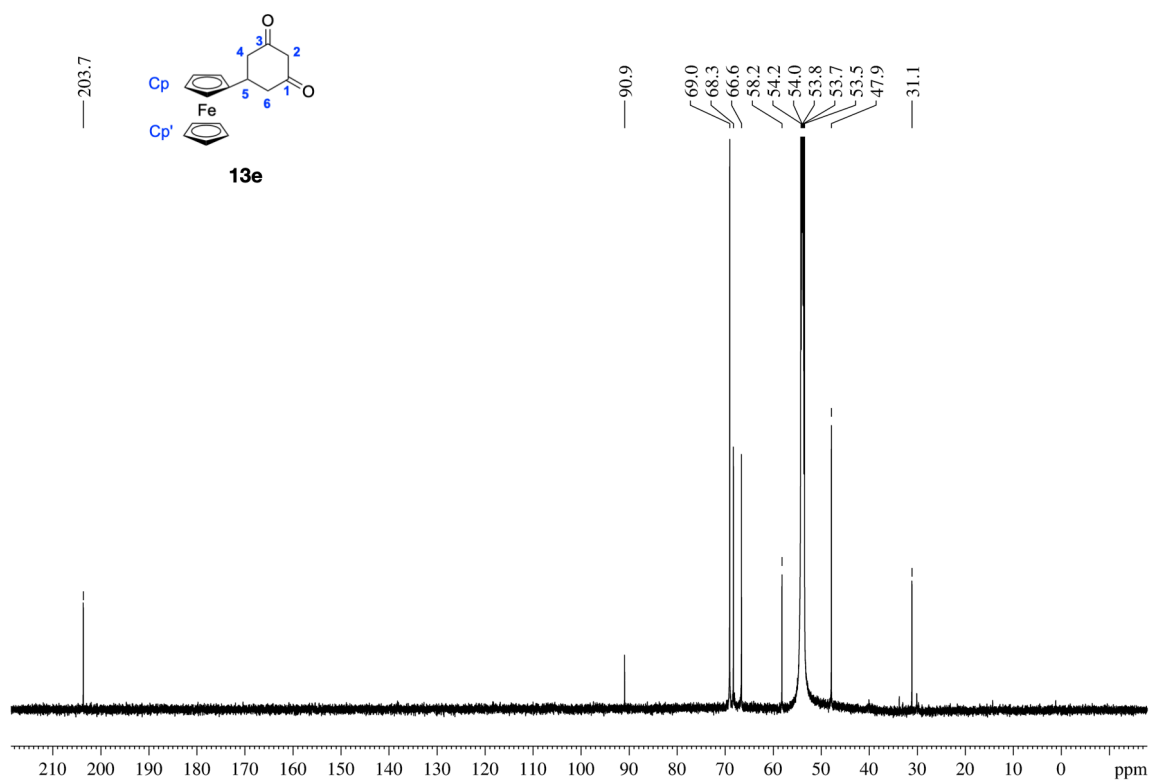


Figure S17. ¹³C{¹H} NMR spectrum of **13e** in DMSO-d₆
CD₂Cl₂ - 243K

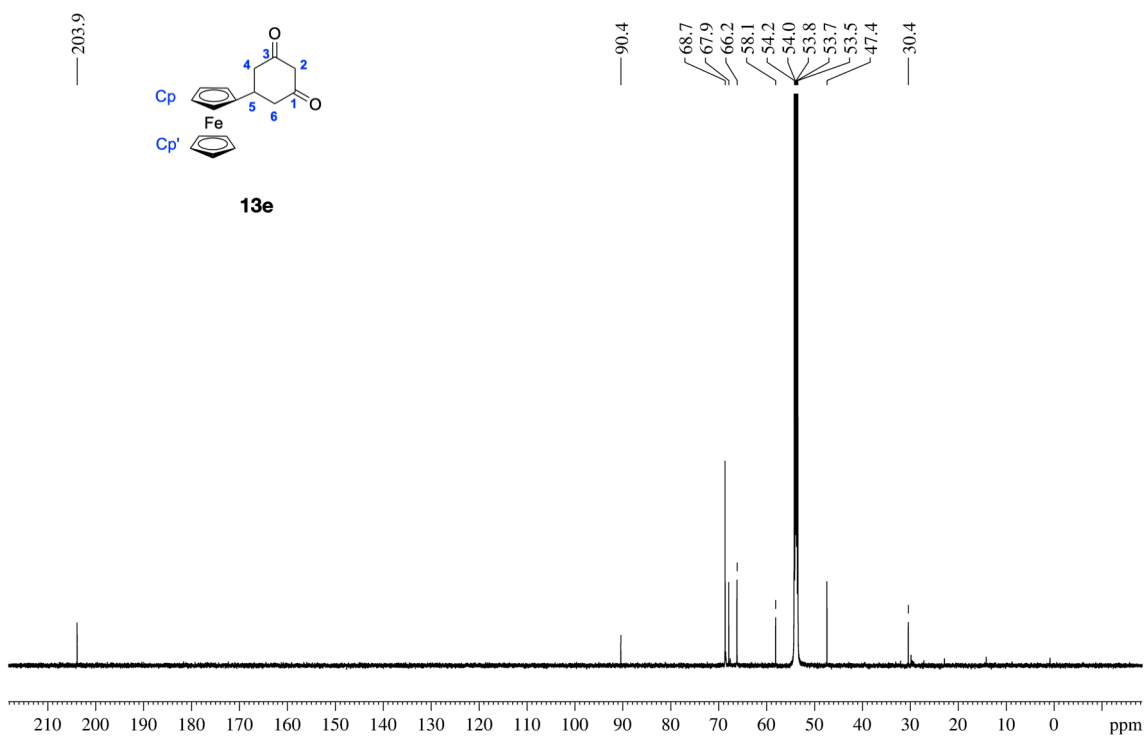


Figure S18. ¹³C{¹H} NMR spectrum of **13e** in DMSO-d₆

Sample Information
 Sample Type : Unknown
 Sample Name : DPX-90010
 Sample ID : DPX-90010
 Vial# : 1
 Injection Volume : 0.3

Method
 Time : 4.00
 7.00
 7.05
 13.00

Module
 Pumps
 Pumps
 Pumps
 Controller

Command
 B.Conc
 B.Conc
 B.Conc
 Stop

<<Column Information>>
 Column Name : XB-C18
 Column Length : 50 mm
 Inner Diameter : 2.1 mm

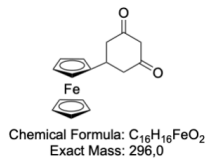
<<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table

Peak#	Ret. Time	Area%
1	8.774	98.505
2	9.294	0.903
3	9.716	0.592
Total		100.000



13e

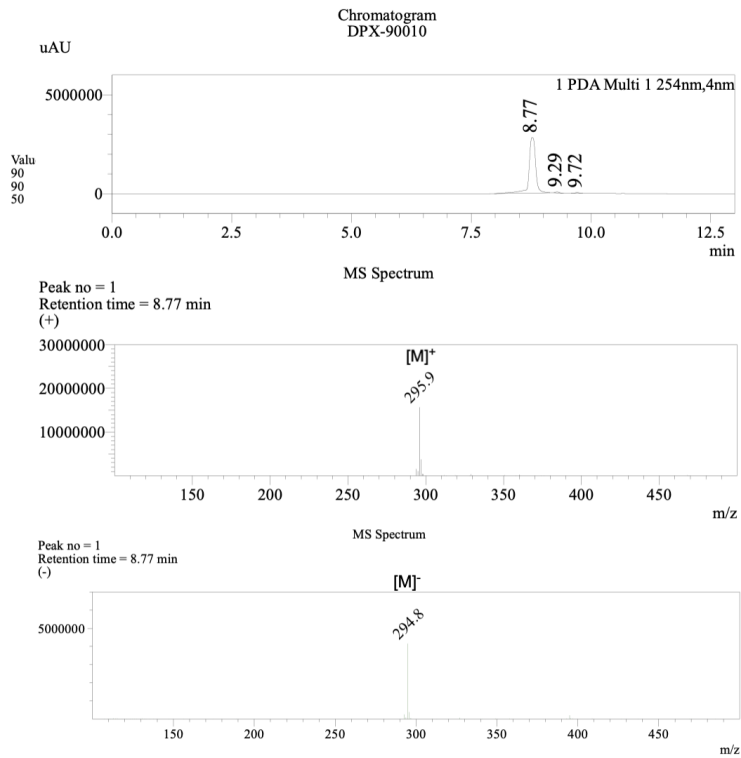


Figure S19. HPLC-MS analysis of 13e

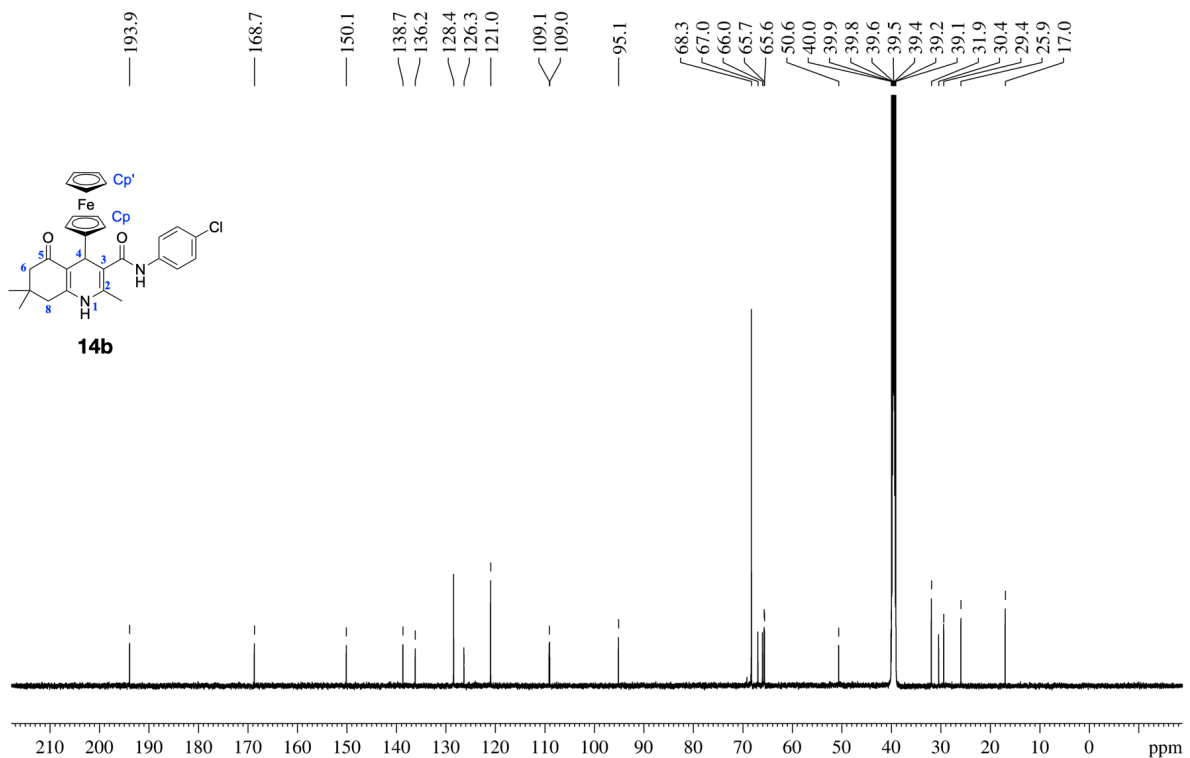


Figure S20. ¹³C{¹H} NMR spectrum of 14b in DMSO-d₆

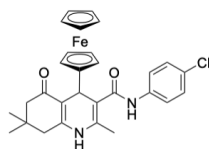
Sample Information
 Sample Type : Unknown
 Sample Name : K GK-00530b
 Sample ID : K GK-00530b
 Vial# : 42
 Injection Volume : 0.3

Method
 <<LC Time Program>>
 Time : 4.00
 7.00
 7.05
 13.00
 Module : Pumps
 Pumps
 Pumps
 Controller
 Command : B.Conc
 B.Conc
 B.Conc
 Stop
 Valu : 90
 90
 50

<<Column Information>>
 Column Name : XB-C18
 Column Length : 50 mm
 Inner Diameter : 2.1 mm
 <<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH
 <<Data Acquisition>>
 LC Stop Time : 13.00 min
 <<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table

Peak#	Ret. Time	Area%
1	6.975	97.965
2	7.827	0.755
3	8.178	0.931
4	8.321	0.349
Total		100.000



Chemical Formula: C₂₉H₂₉ClFeN₄O₂
 Exact Mass: 528.1

14b

Figure S21. ¹³C{¹H} NMR spectrum of **14b** in DMSO-d₆

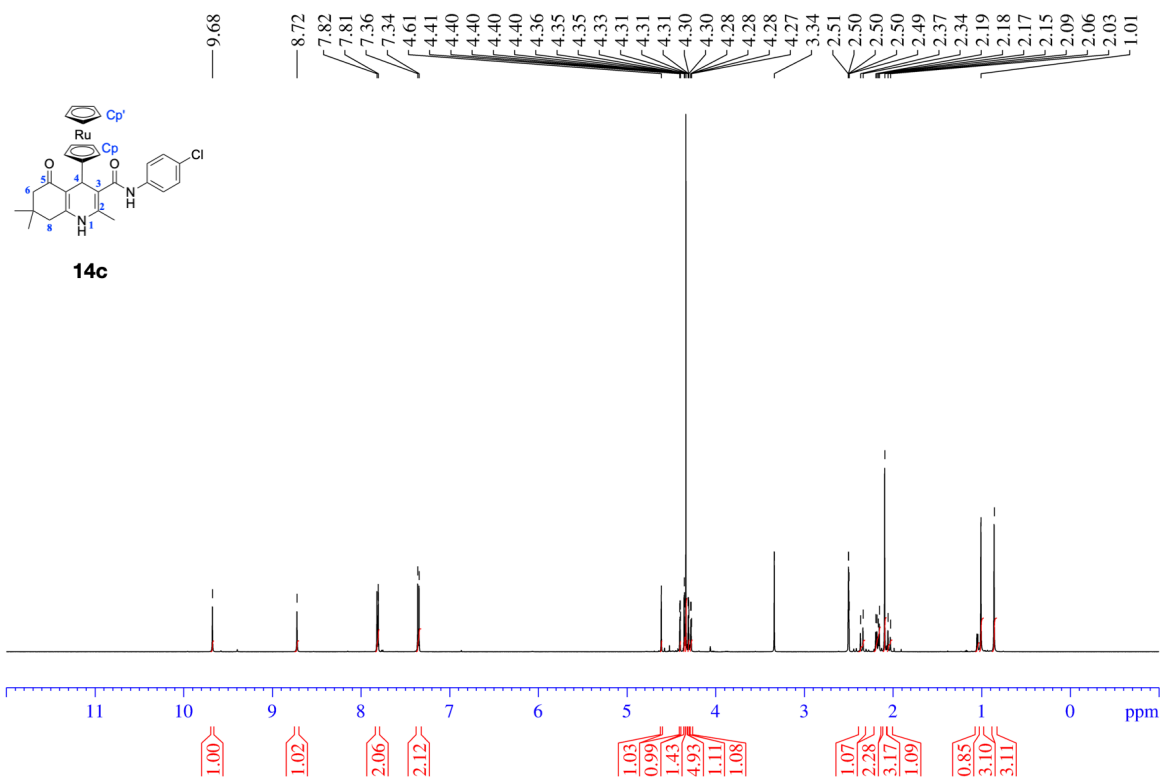
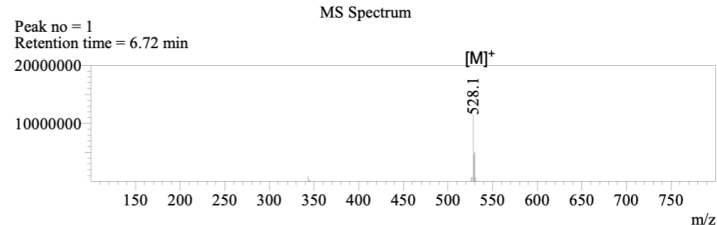
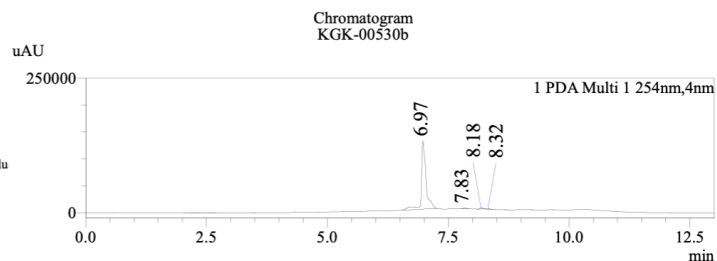


Figure S22. ¹³C{¹H} NMR spectrum of **14c** in DMSO-d₆

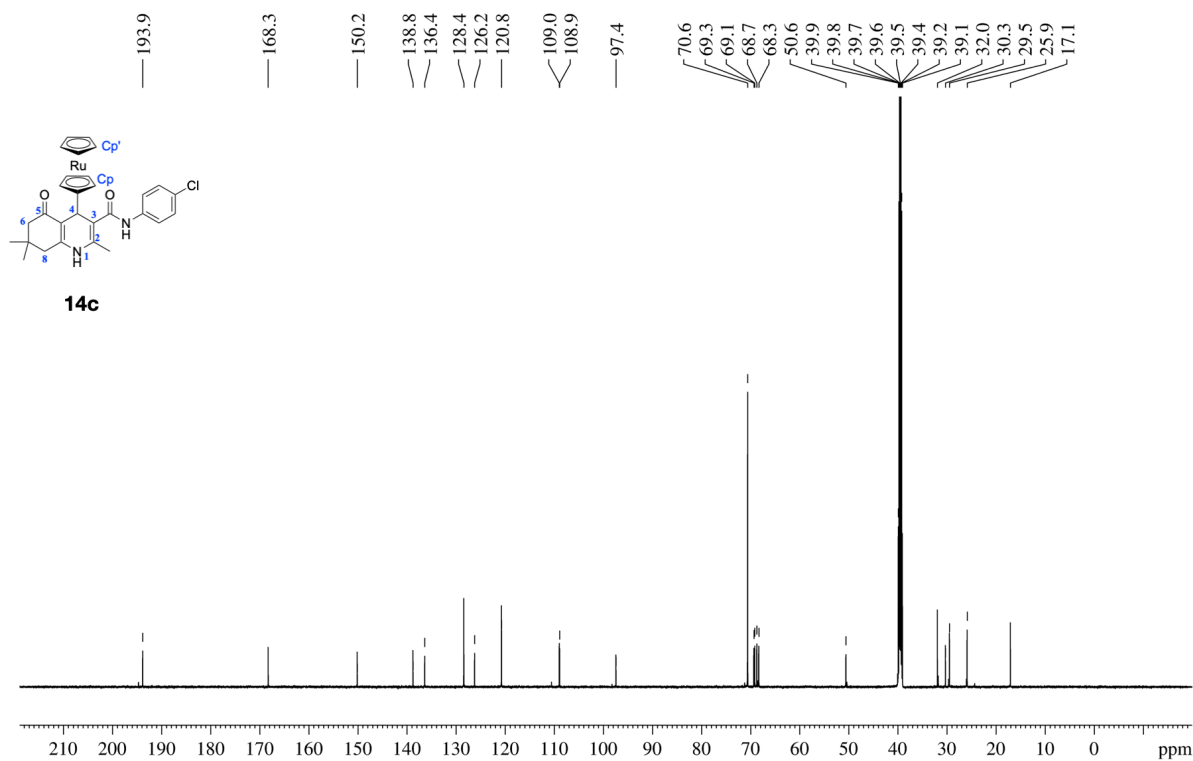


Figure S23. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14c** in $\text{DMSO-}d_6$

Sample Information
 Sample Type : Unknown
 Sample Name : DPX-90033
 Sample ID : DPX-90033
 Vial# : 40
 Injection Volume : 0.3

Method
 <<LC Time Program>>
 Time : 4.00 Module : Pumps Command : B.Conc
 7.00 Pumps B.Conc
 7.05 Pumps B.Conc
 13.00 Controller Stop

<<Column Information>>
 Column Name : XB-C18
 Column Length : 50 mm
 Inner Diameter : 2.1 mm

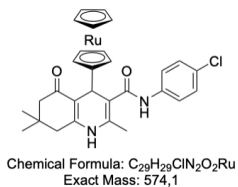
<<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

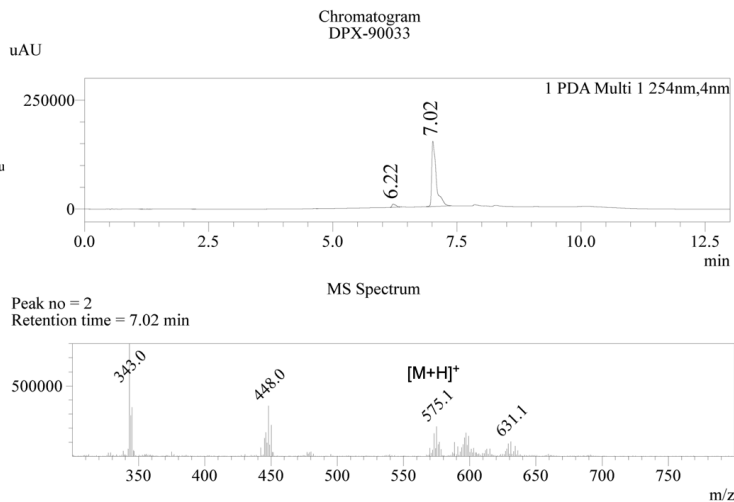
Peak Table

Peak#	Ret. Time	Area%
1	6.215	3.913
2	7.016	96.087
Total		100.000



14c

Figure S24. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14c** in $\text{DMSO-}d_6$



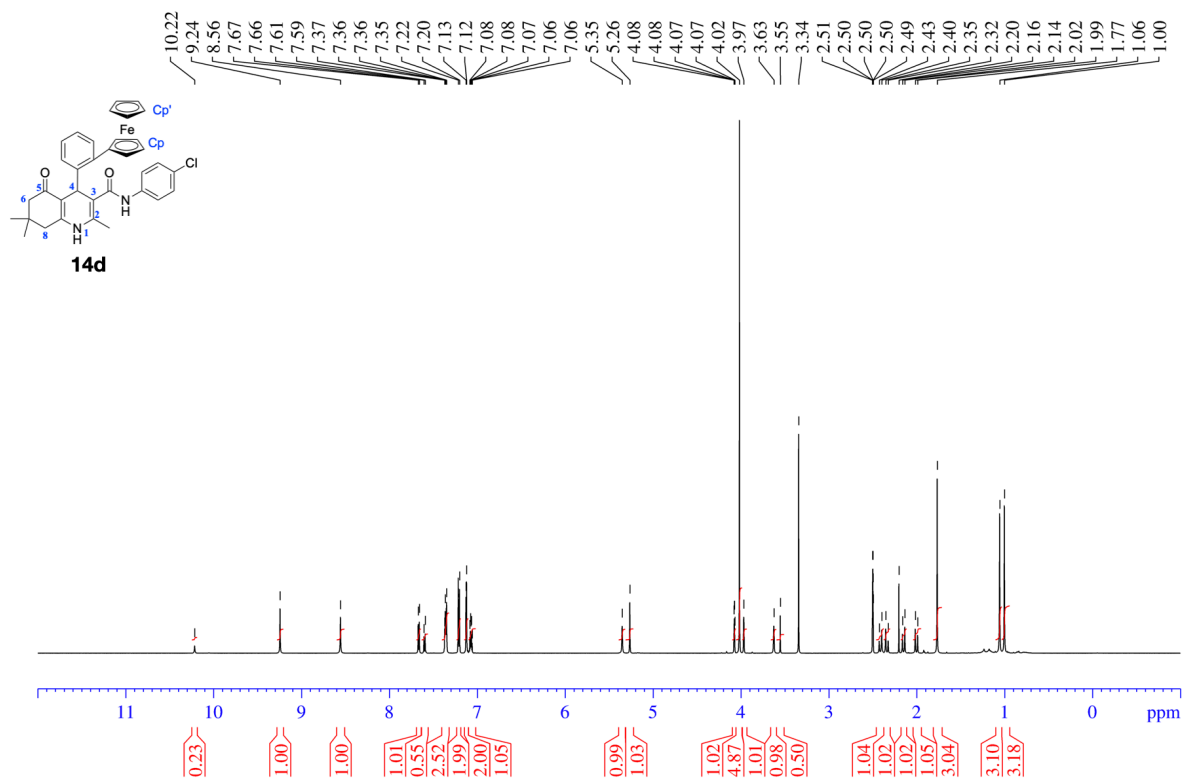


Figure S25. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14d** in DMSO-d_6

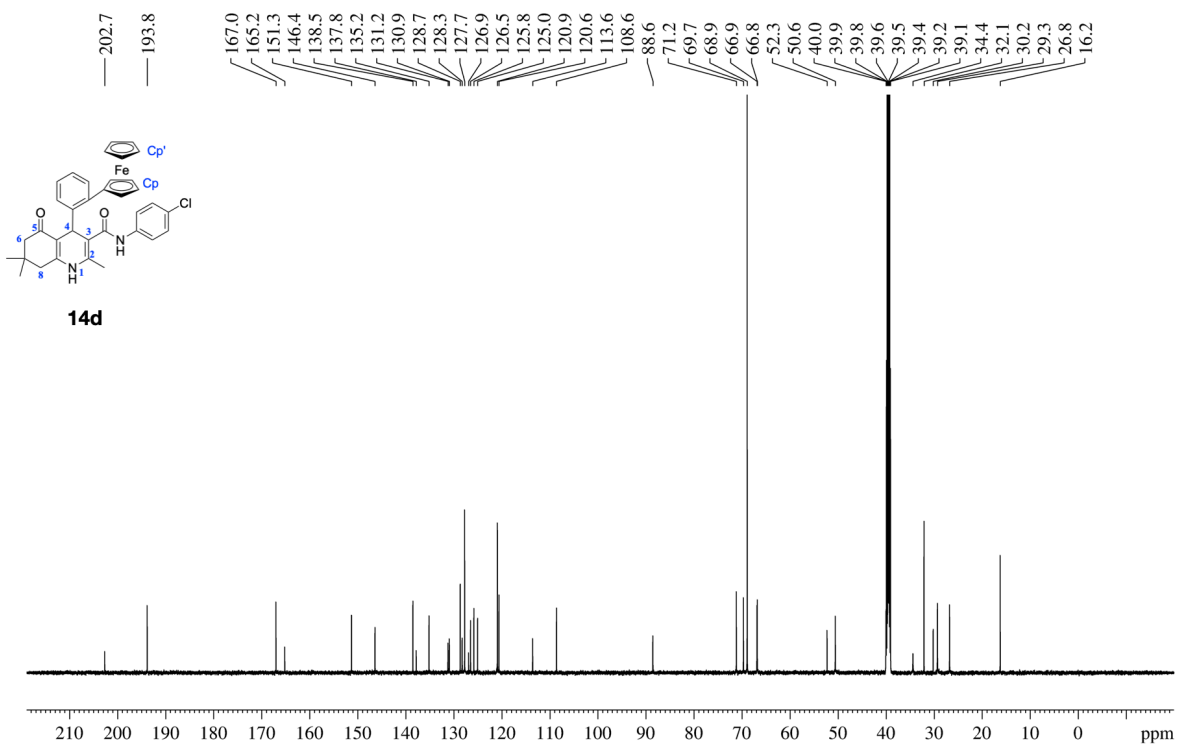


Figure S26. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14d** in DMSO-d_6

Sample Information
 Sample Type : Unknown
 Sample Name : kgk-00526
 Sample ID : kgk-00526
 Vial# : 2
 Injection Volume : 0.3

Method
 <<LC Time Program>>
 Time : 13.00
 Module : Pumps
 Pumps : B.Conc
 Controller : Stop

<<Column Information>>
 Column Name : XB-C18
 Column Length : 50 mm
 Inner Diameter : 2.1 mm

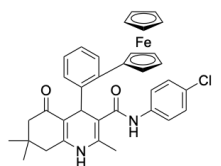
<<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table

Peak#	Ret. Time	Area%
1	10.206	11.888
2	10.442	88.112
Total		100.000



Chemical Formula: C₃₅H₃₃ClFeN₂O₂
 Exact Mass: 604.2

14d

Figure S27. ¹³C{¹H} NMR spectrum of 14d in DMSO-d₆

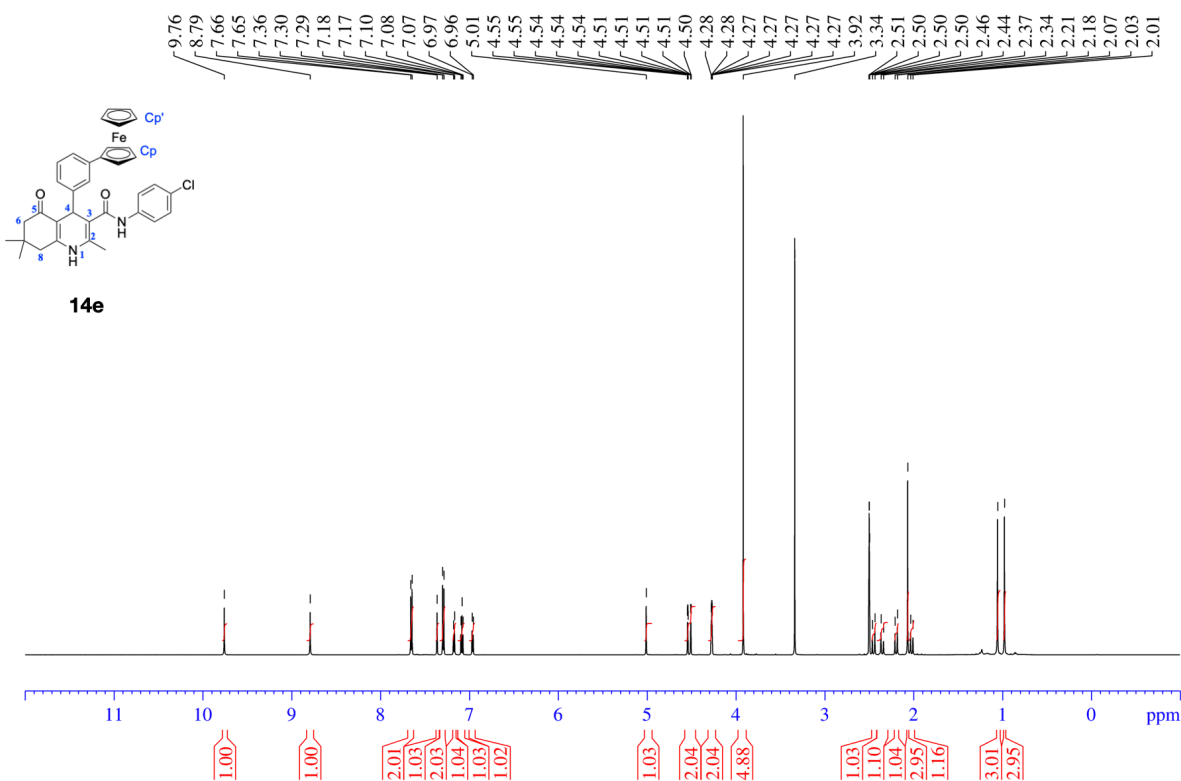
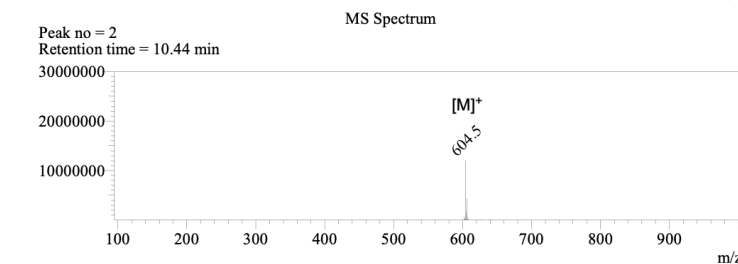
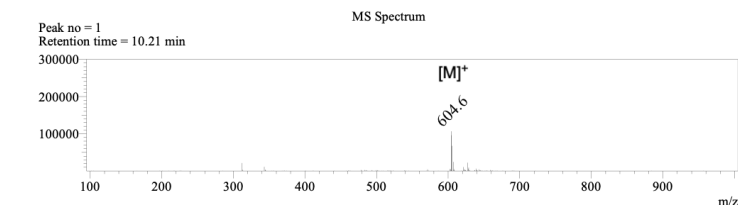
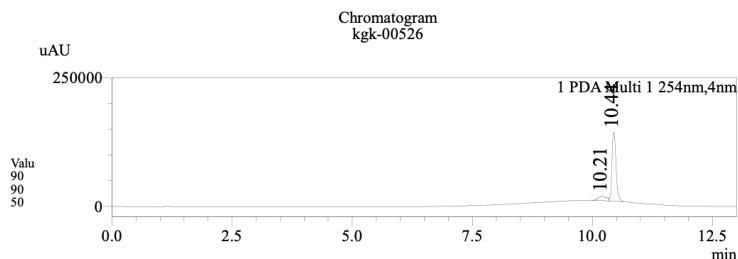


Figure S28. ¹³C{¹H} NMR spectrum of 14e in DMSO-d₆

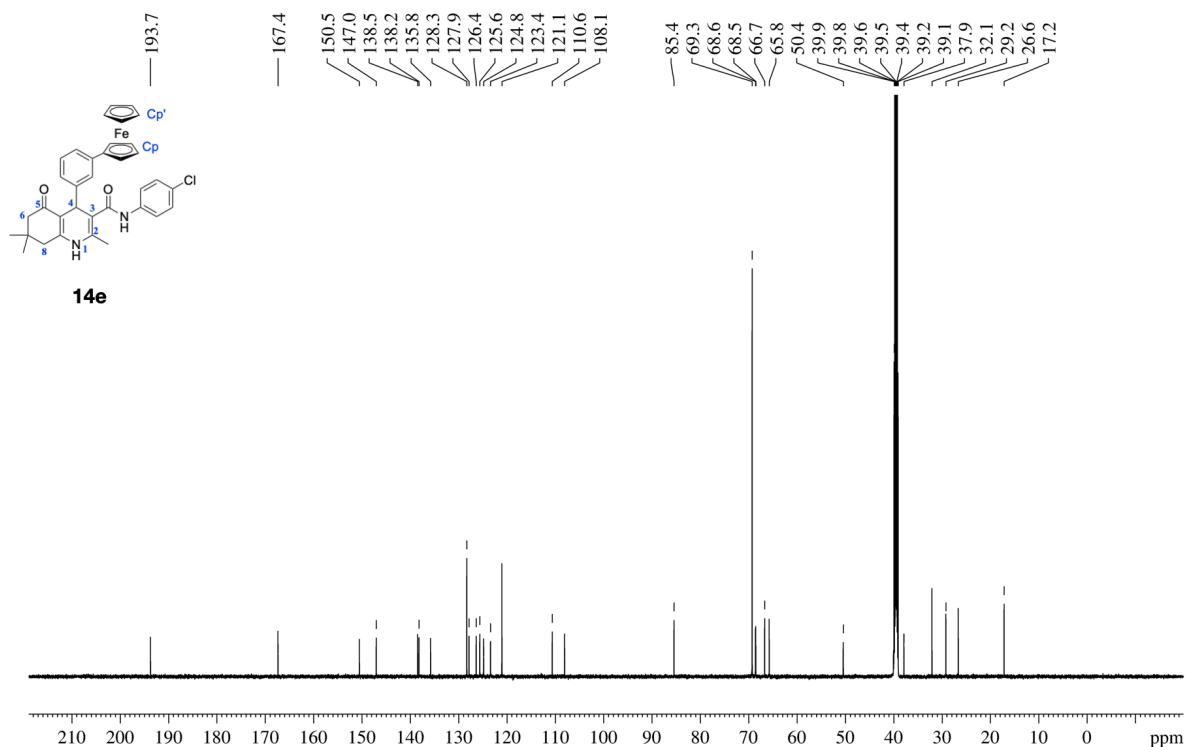


Figure S29. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14e** in DMSO-d_6

Sample Information

Sample Type : Unknown
 Sample Name : KGK-00525
 Sample ID : KGK-00525
 Vial# : 78
 Injection Volume : 10

Method

Time	Module	Command
4.00	Pumps	B.Conc
7.00	Pumps	B.Conc
7.00	Pumps	B.Conc
13.00	Controller	Stop

<<Mobile Phase Name>>
 Mobile Phase A : water+0.01% HCOOH
 Mobile Phase B : Methanol+0.01% HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table

Peak#	Ret. Time	Area%
1	8.524	8.786
2	8.658	91.214
Total		100.000

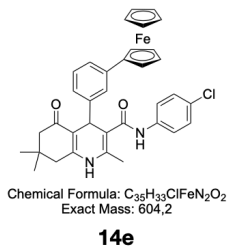
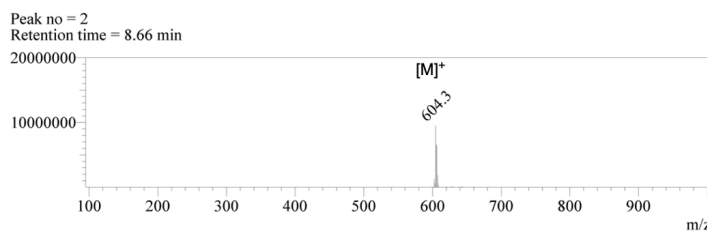
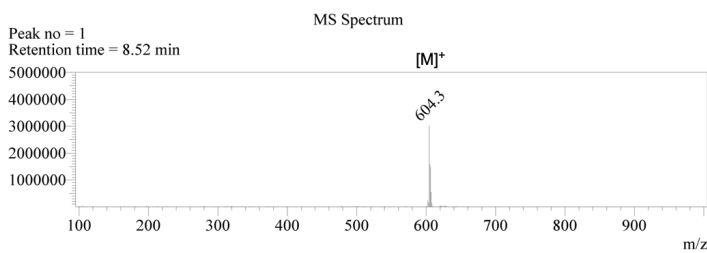
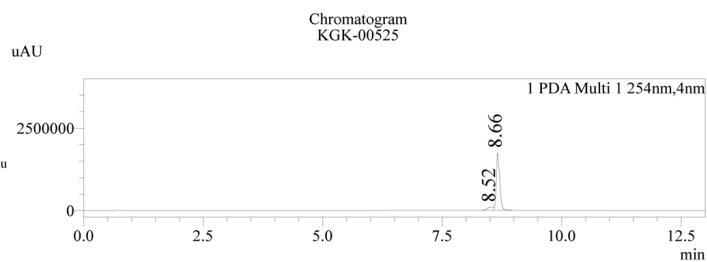


Figure S30. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14e** in DMSO-d_6

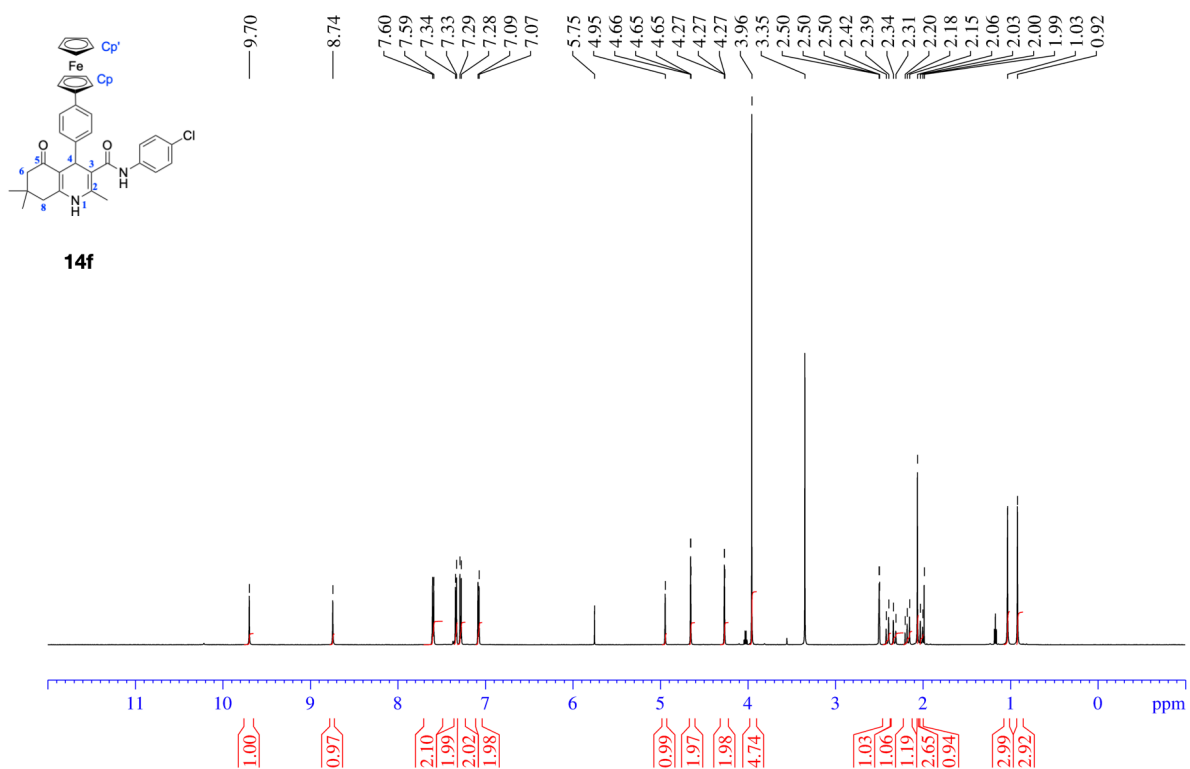


Figure S31. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14f** in DMSO-d_6

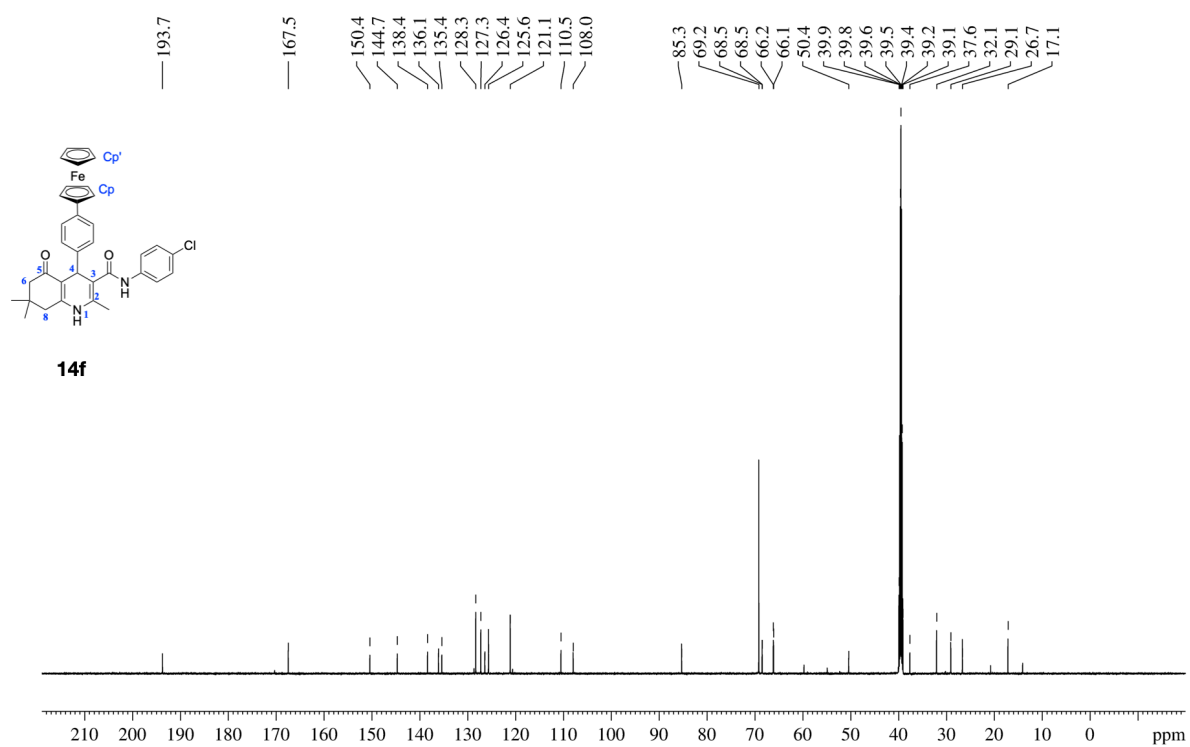


Figure S32. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14f** in DMSO-d_6

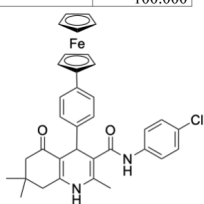
Sample Information
 Sample Type : Unknown
 Sample Name : KGK-00524
 Sample ID : KGK-00524
 Vial# : 44
 Injection Volume : 0.3

Method
 <<LC Time Program>>
 Time : 13.00
 Module : Pumps
 Pumps : Pumps
 Controller : Controller
 Command : B.Conc
 B.Conc : B.Conc
 B.Conc : B.Conc
 Stop : Stop

<<Column Information>>
 Column Name : XB-C18
 Column Length : 50 mm
 Inner Diameter : 2.1 mm
 <<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH
 <<Data Acquisition>>
 LC Stop Time : 13.00 min
 <<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table

Peak#	Ret. Time	Area%
1	7.265	7.816
2	7.483	92.184
Total		100.000



Chemical Formula: C₃₅H₃₅ClFeN₂O₂
 Exact Mass: 604.2

14f

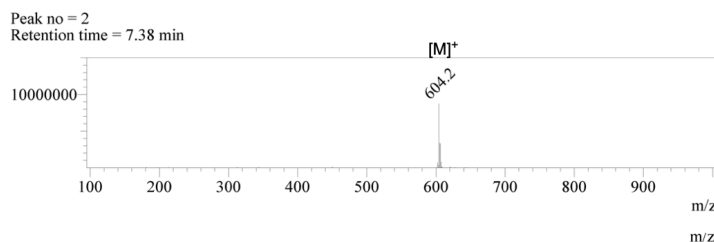
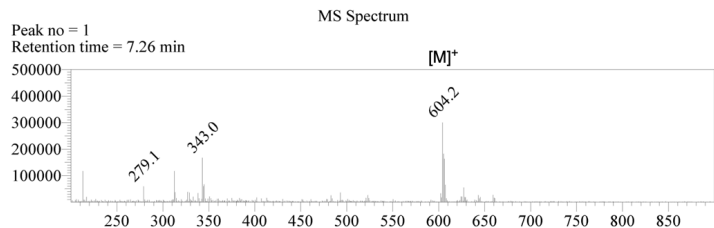
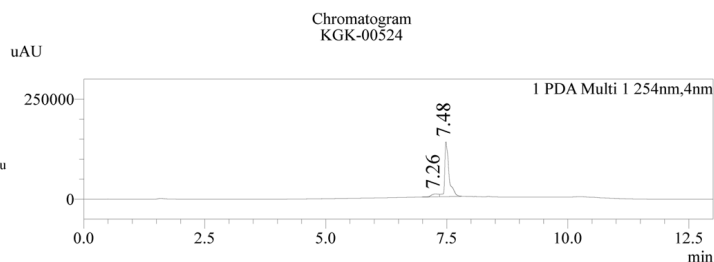


Figure S33. ¹³C{¹H} NMR spectrum of **14f** in DMSO-d₆

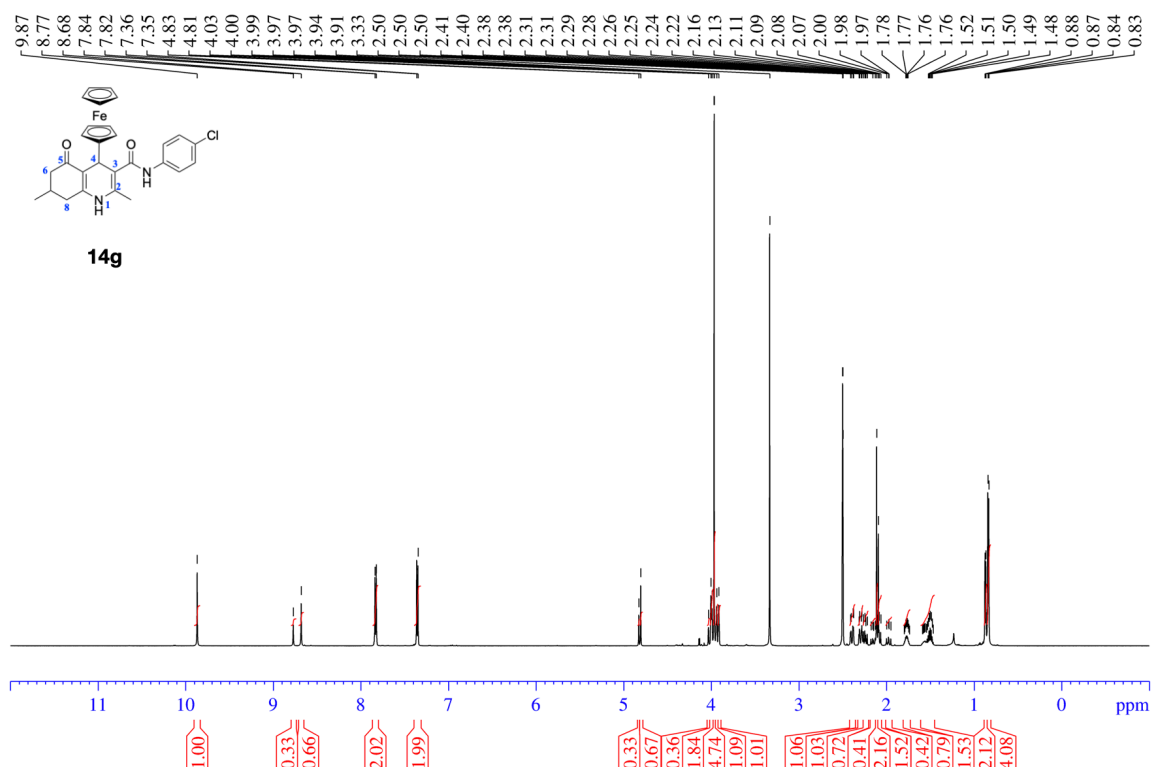


Figure S34. ¹³C{¹H} NMR spectrum of **14g** in DMSO-d₆

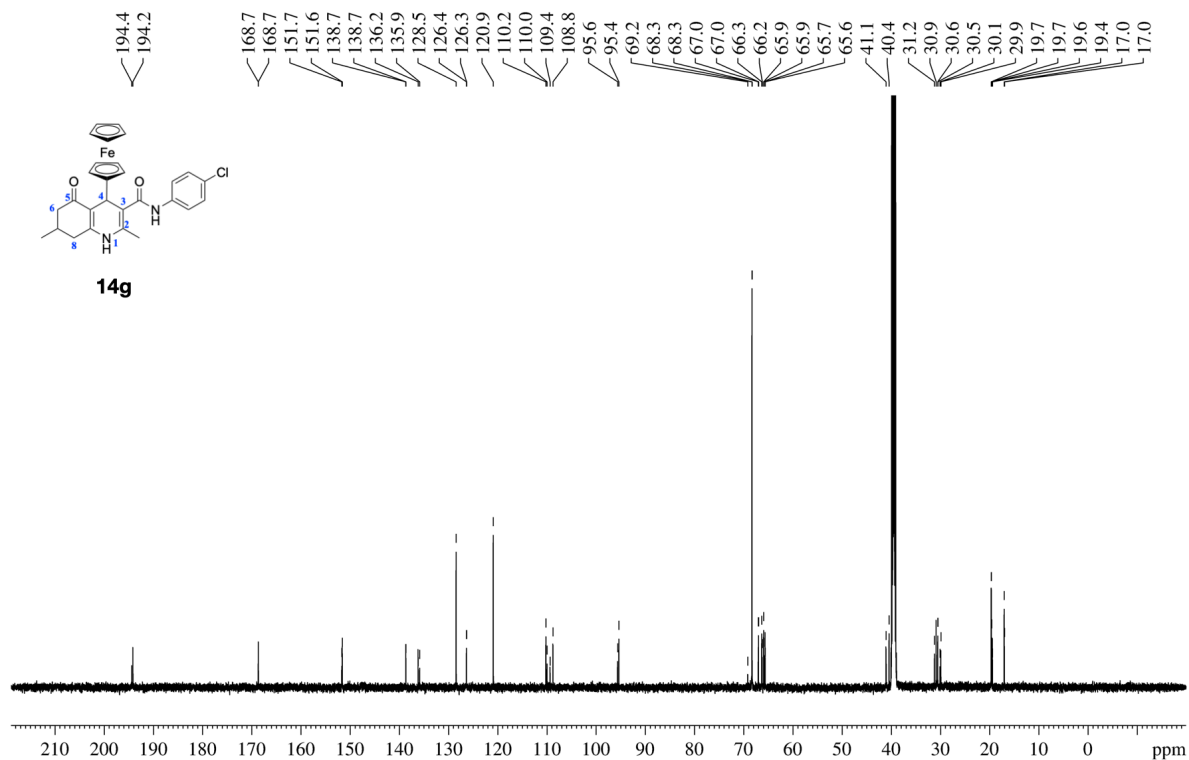


Figure S35. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14g** in $\text{DMSO-}d_6$

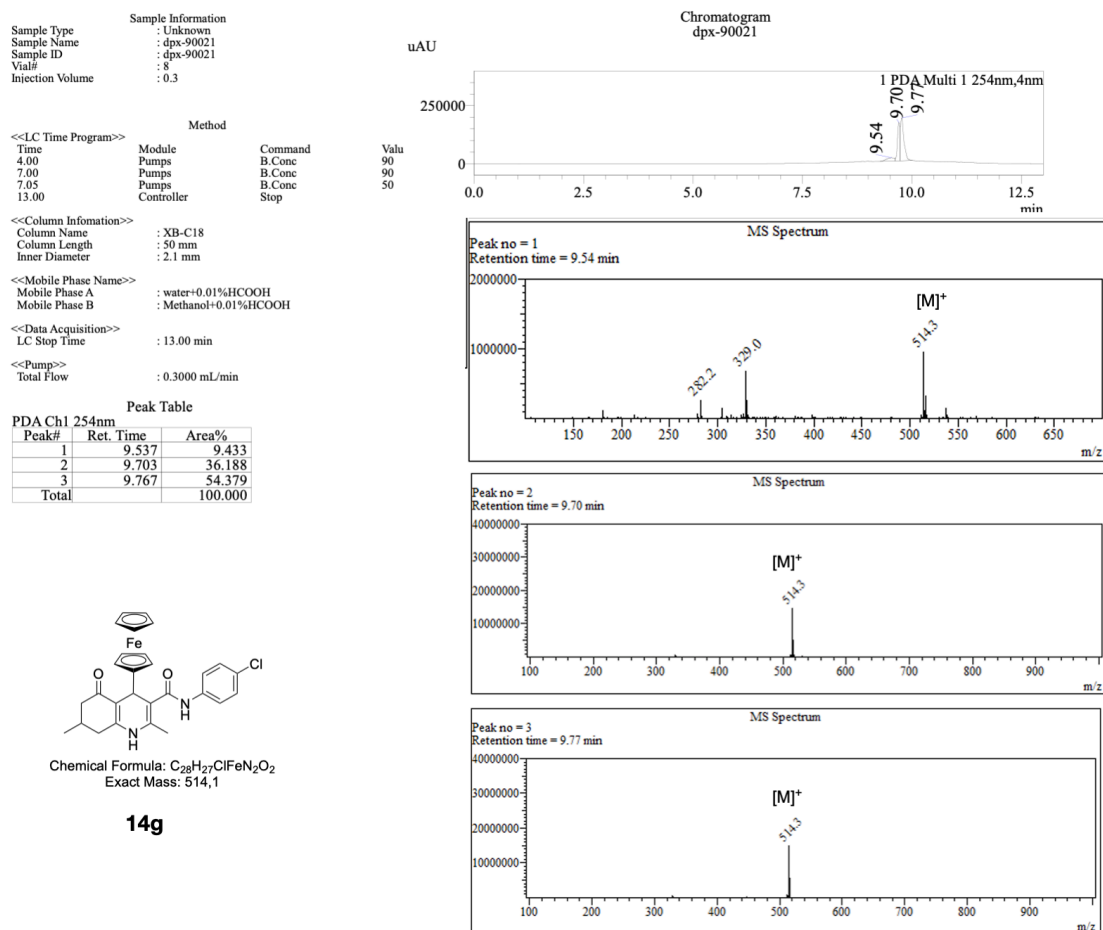


Figure S36. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14g** in $\text{DMSO-}d_6$

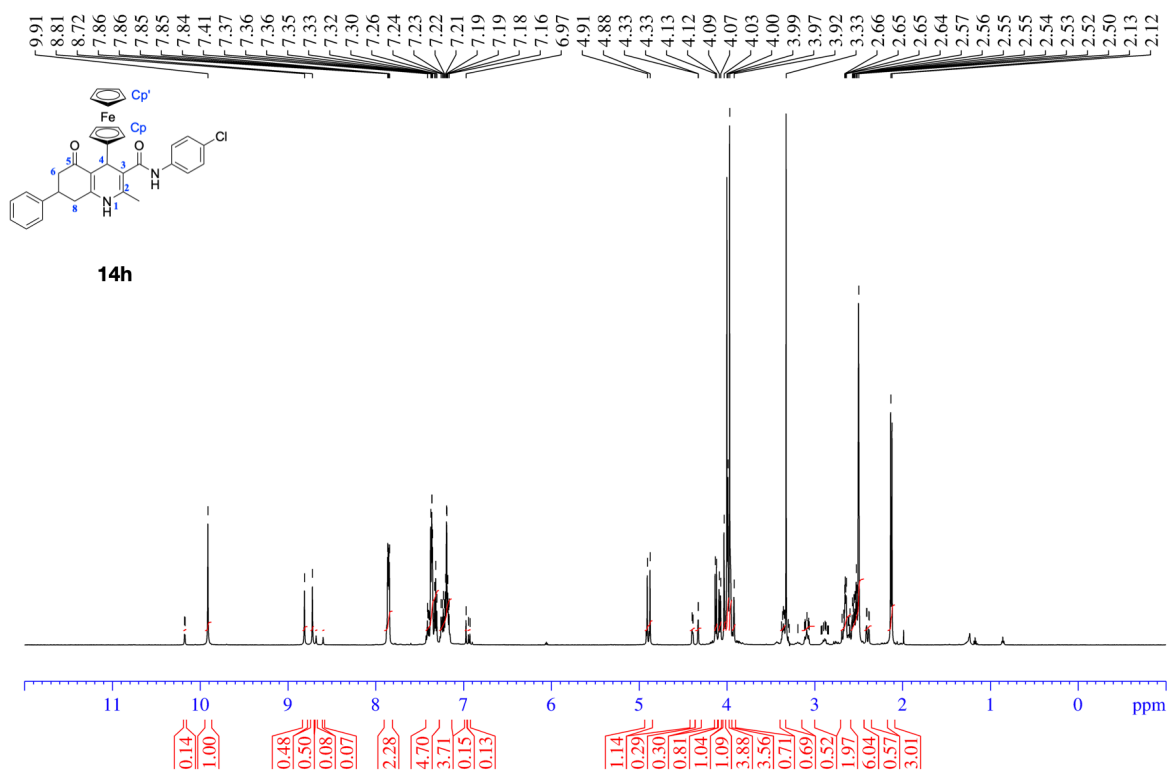


Figure S37. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14h** in DMSO- d_6

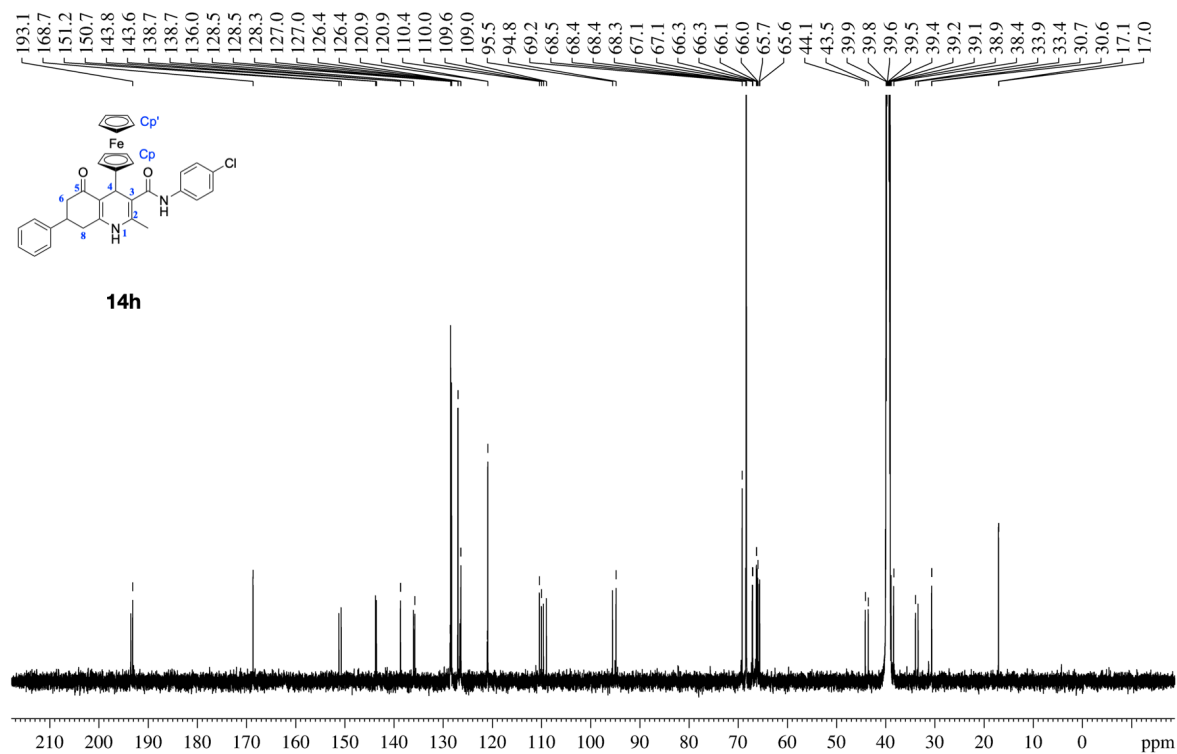


Figure S38. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14h** in DMSO- d_6

Sample Information
 Sample Type : Unknown
 Sample Name : DPX-90024pwt
 Sample ID : DPX-90024pwt
 Vial# : 90
 Injection Volume : 0.5

Method
 <<LC Time Program>>
 Time : 13.00
 Module : Pumps
 Command : B.Conc
 Pumps : B.Conc
 Pumps : B.Conc
 Controller : Stop

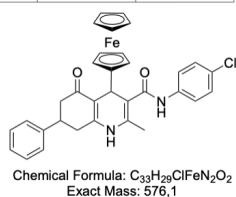
<<Column Information>>
 Column Name : XB-C18
 Column Length : 50 mm
 Inner Diameter : 2.1 mm

<<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak#	Ret. Time	Area%
1	7.477	7.797
2	7.614	40.424
3	7.713	51.779
Total		100.000



14h

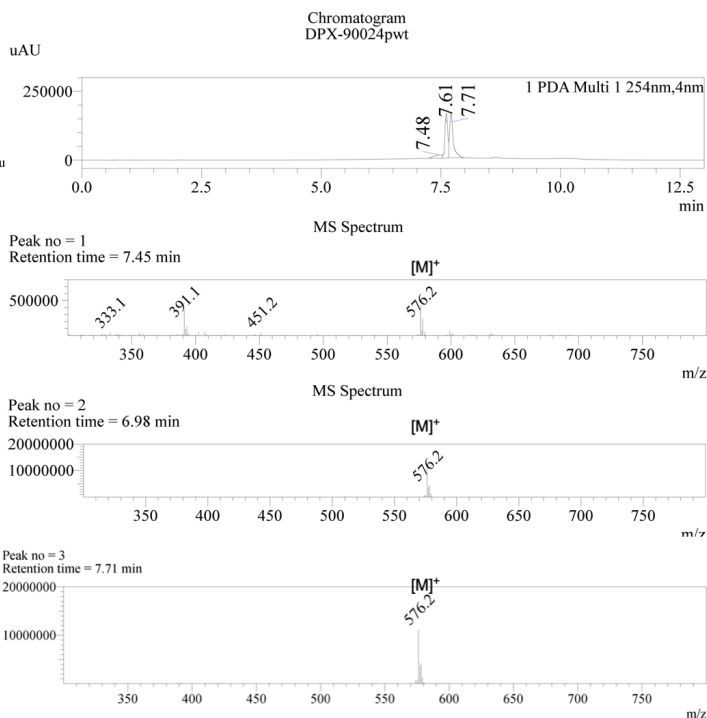


Figure S39. ¹³C{¹H} NMR spectrum of 14h in DMSO-d₆

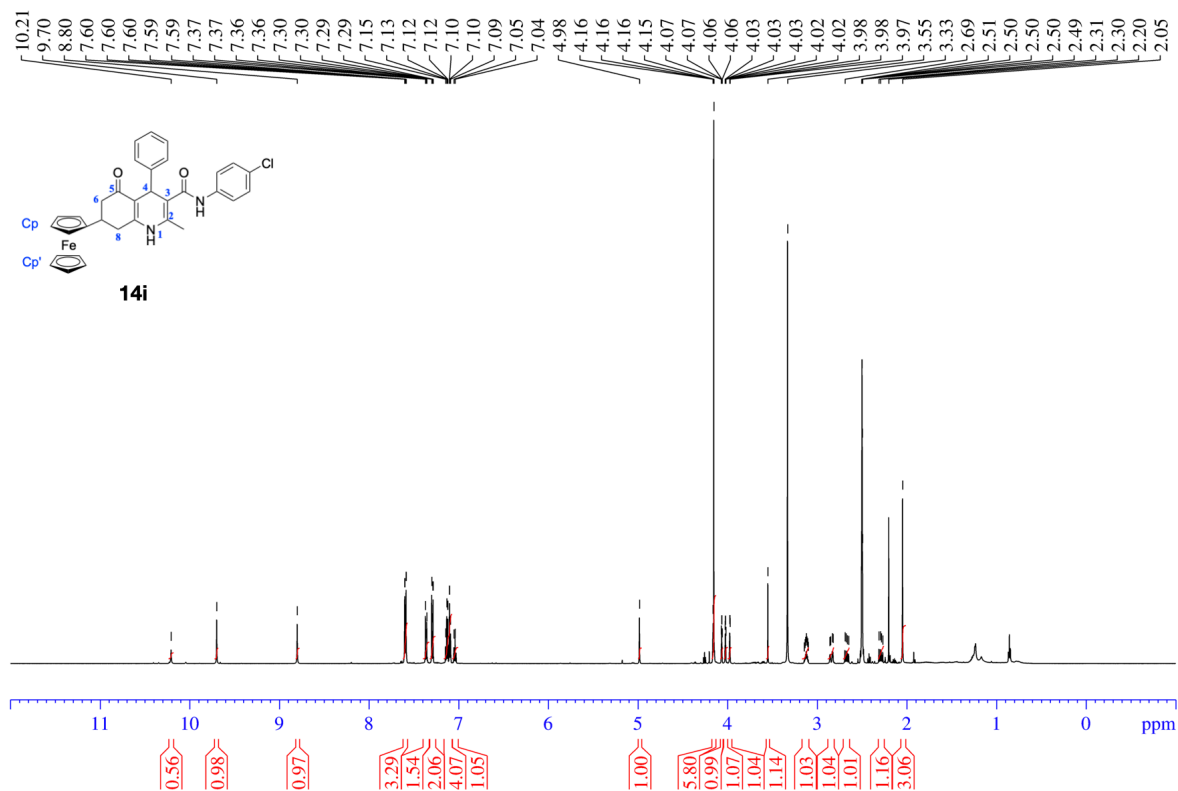


Figure S40. ¹³C{¹H} NMR spectrum of 14i in DMSO-d₆

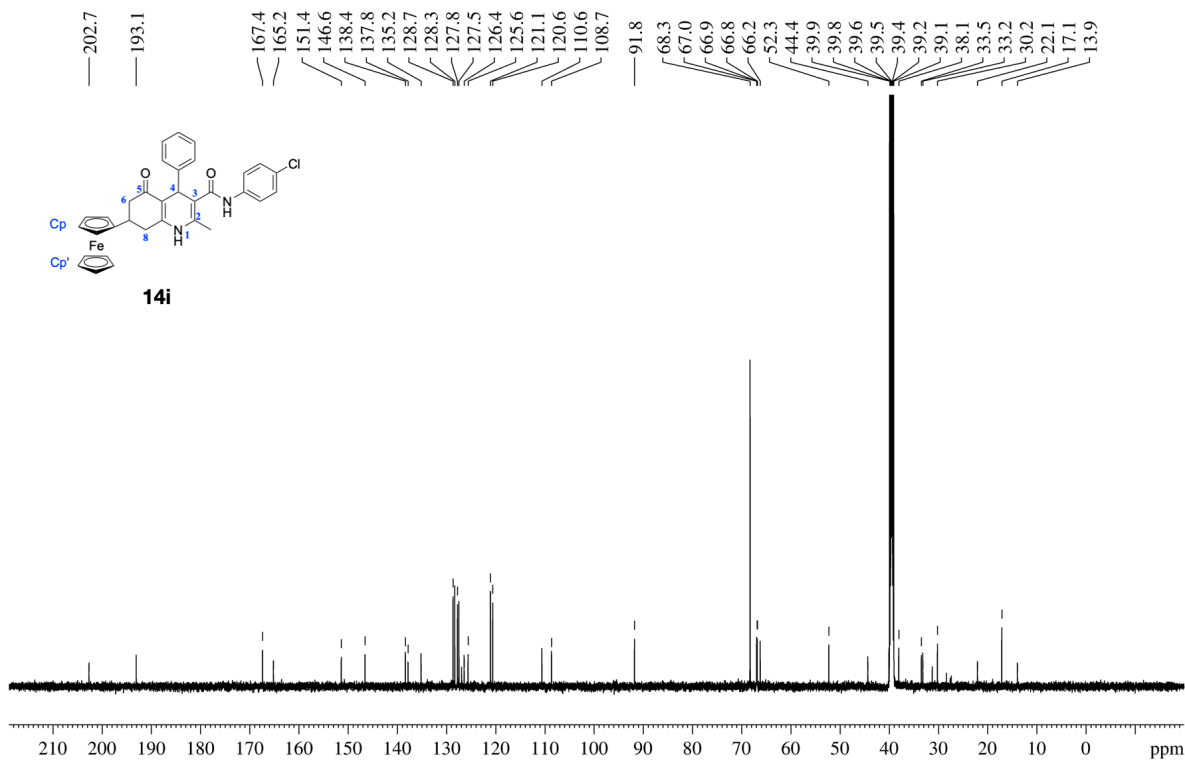


Figure S41. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14i** in DMSO-d_6

Sample Information
 Sample Type : Unknown
 Sample Name : kgk-90026_MeOH_5-9-5
 Sample ID : kgk-90026_MeOH_5-9-3
 Vial# : 103
 Injection Volume : 0.5

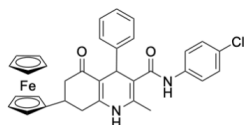
Method
 <<LC Time Program>>
 Time : 4.00, 7.00, 7.05, 13.00
 Module : Pumps, Pumps, Pumps, Controller
 Command : B.Conc, B.Conc, B.Conc, Stop

Mobile Phase Name
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH

Data Acquisition
 LC Stop Time : 13.00 min

Pump
 Total Flow : 0.3000 mL/min

Peak Table		
Peak#	Ret. Time	Area%
1	7.911	100.000
Total		100.000



Chemical Formula: $\text{C}_{33}\text{H}_{29}\text{ClFeN}_2\text{O}_2$
 Exact Mass: 576.1

14i

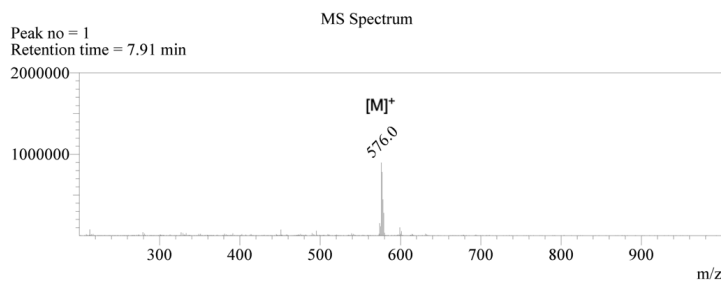
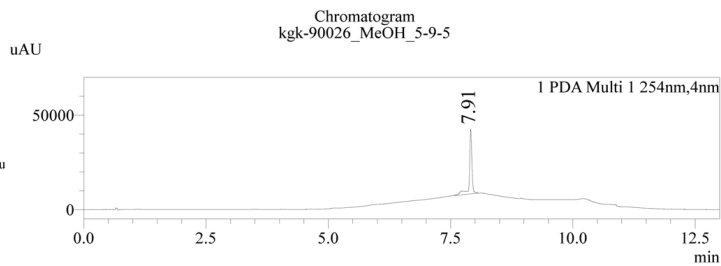


Figure S42. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14i** in DMSO-d_6

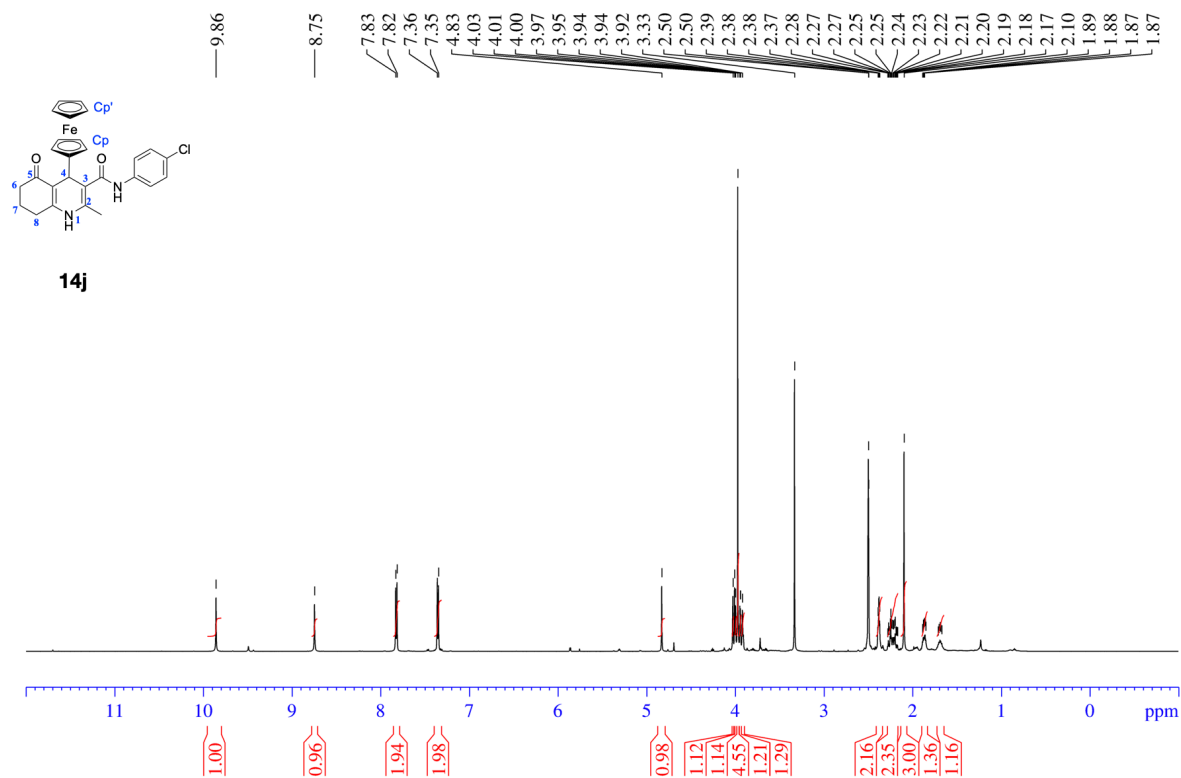


Figure S43. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14j** in DMSO-d_6

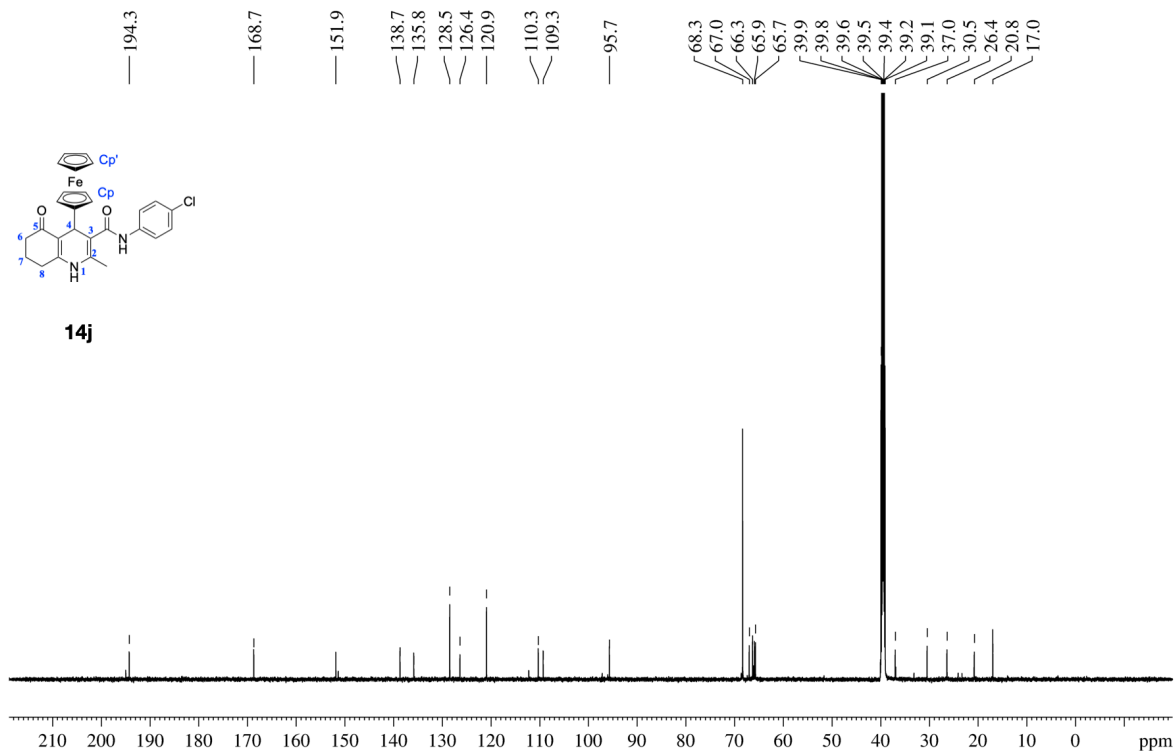


Figure S44. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **14j** in DMSO-d_6

Sample Information
 Sample Type : Unknown
 Sample Name : dhp-90025_pwt dp
 Sample ID : dhp-90025_pwt dp
 Vial# : 15
 Injection Volume : 0.3

Method
 <<LC Time Program>>
 Time : 4.00
 7.00
 7.05
 13.00
 Module : Pumps
 Pumps
 Pumps
 Controller
 Command : B.Conc
 B.Conc
 B.Conc
 Stop
 V
 9
 9
 5

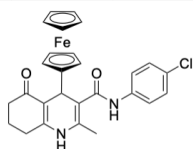
<<Column Information>>
 Column Name : XB-C18
 Column Length : 50 mm
 Inner Diameter : 2.1 mm
 <<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table

Peak#	Ret. Time	Area%
1	5.034	2.217
2	7.189	95.164
3	8.389	2.619
Total		100.000



Chemical Formula: C₂₇H₂₅ClFeN₂O₂
 Exact Mass: 500,1

14j

Chromatogram
 dhp-90025_pwt dp

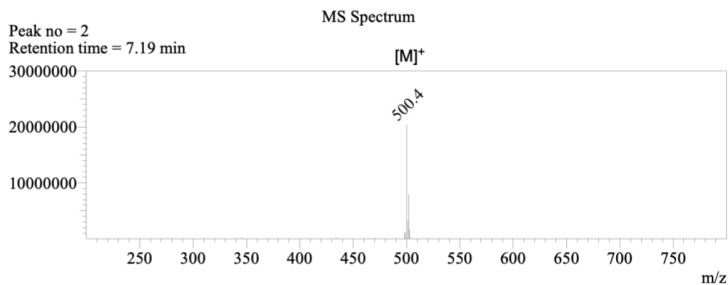
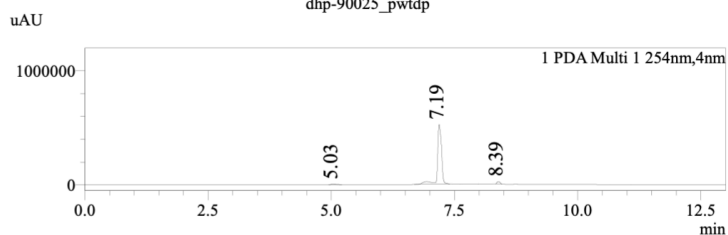


Figure S45. ¹³C{¹H} NMR spectrum of 14j in DMSO-d₆

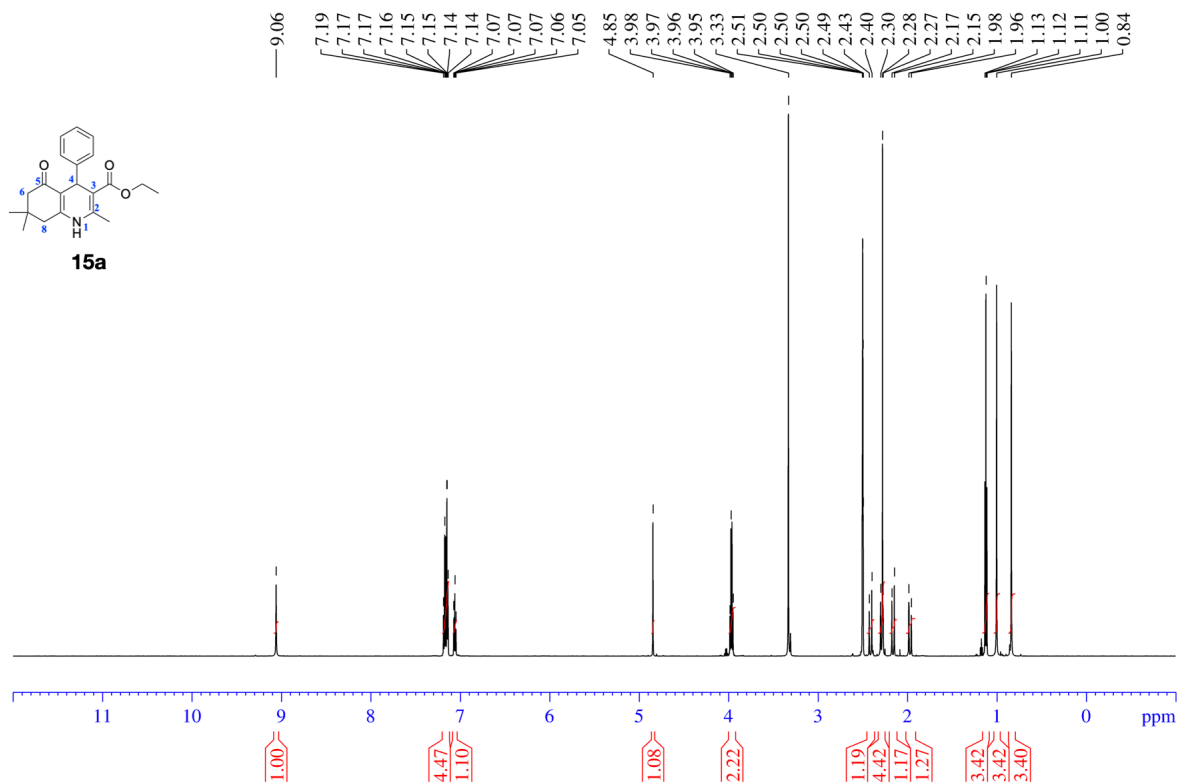


Figure S46. ¹³C{¹H} NMR spectrum of 15a in DMSO-d₆

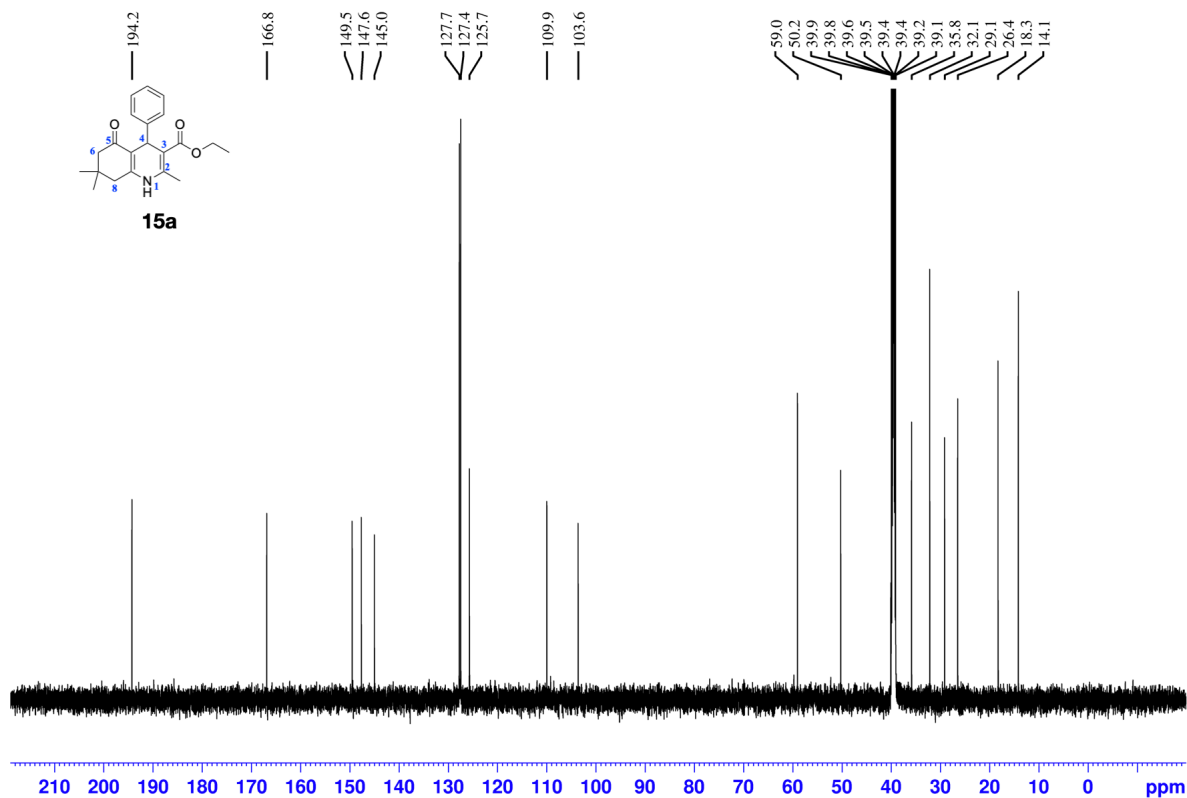


Figure S47. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15a** in DMSO-d_6

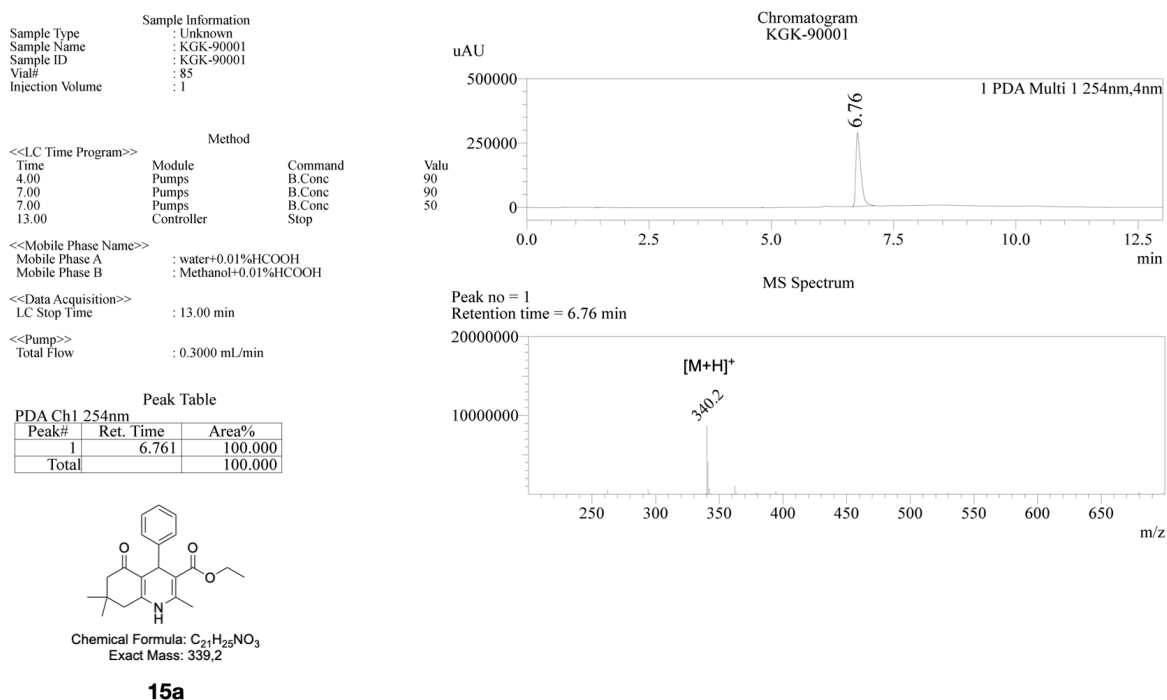


Figure S48. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15a** in DMSO-d_6

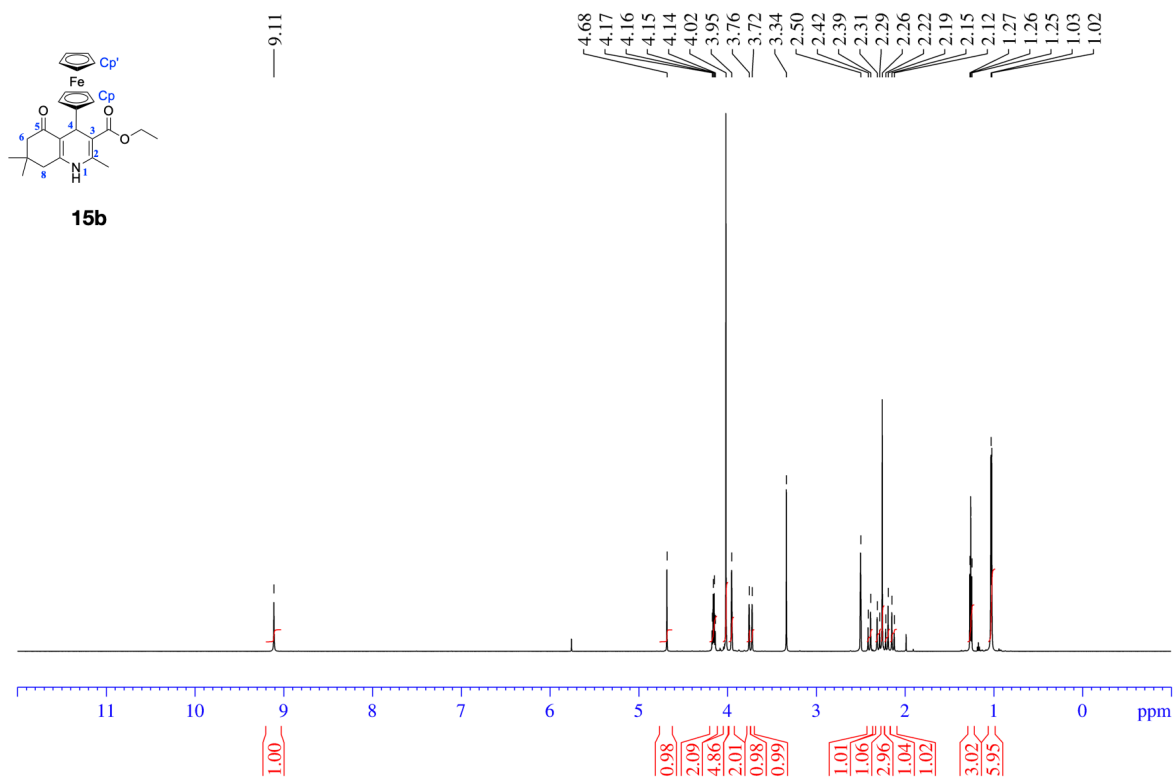


Figure S49. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15b** in DMSO-d_6

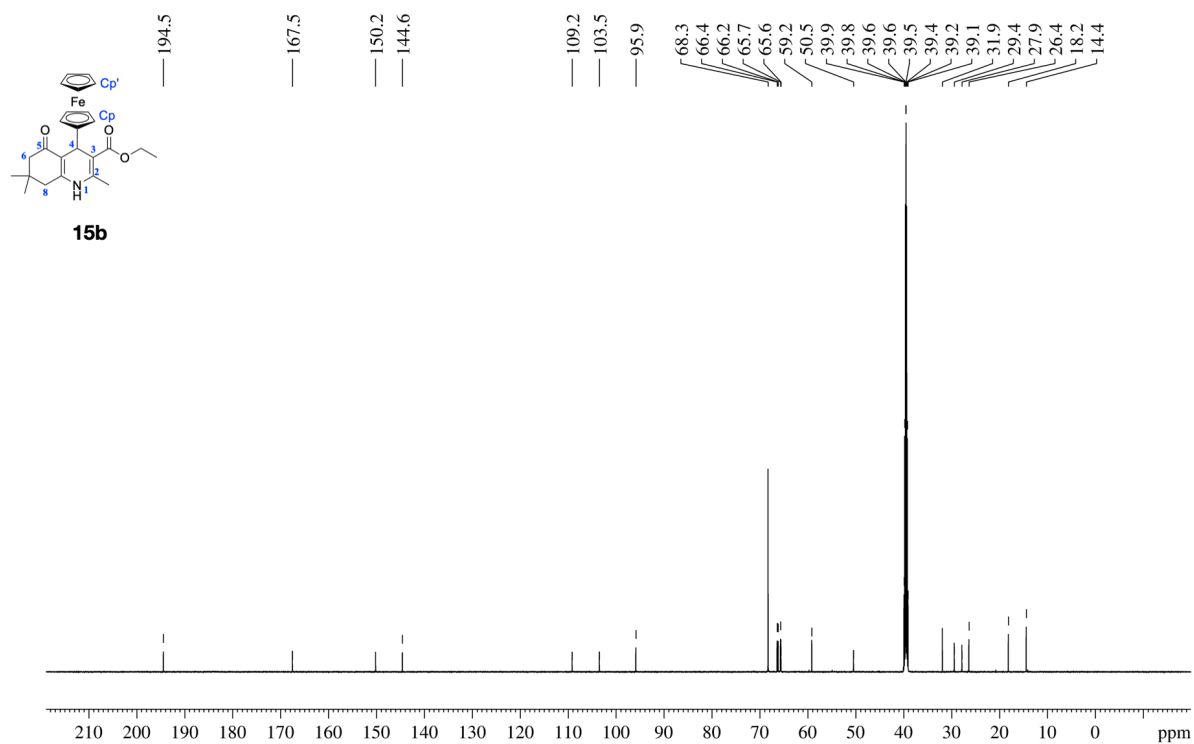


Figure S50. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15b** in DMSO-d_6

Sample Information
 Sample Type : Unknown
 Sample Name : kgk-00529
 Sample ID : kgk-00529a
 Vial# : 102
 Injection Volume : 0.3

Method
 <<LC Time Program>>
 Time : 13.00
 Module : Pumps
 Command : B.Conc
 Pumps : B.Conc
 Pumps : B.Conc
 Controller : Stop

<<Column Information>>
 Column Name : XB-C18
 Column Length : 50 mm
 Inner Diameter : 2.1 mm

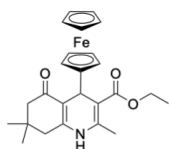
<<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table

Peak#	Ret. Time	Area%
1	6.412	100.000
Total		100.000



Chemical Formula: C₂₅H₂₉FeNO₃
 Exact Mass: 447,1

15b

Figure S51. ¹³C{¹H} NMR spectrum of **15b** in DMSO-d₆

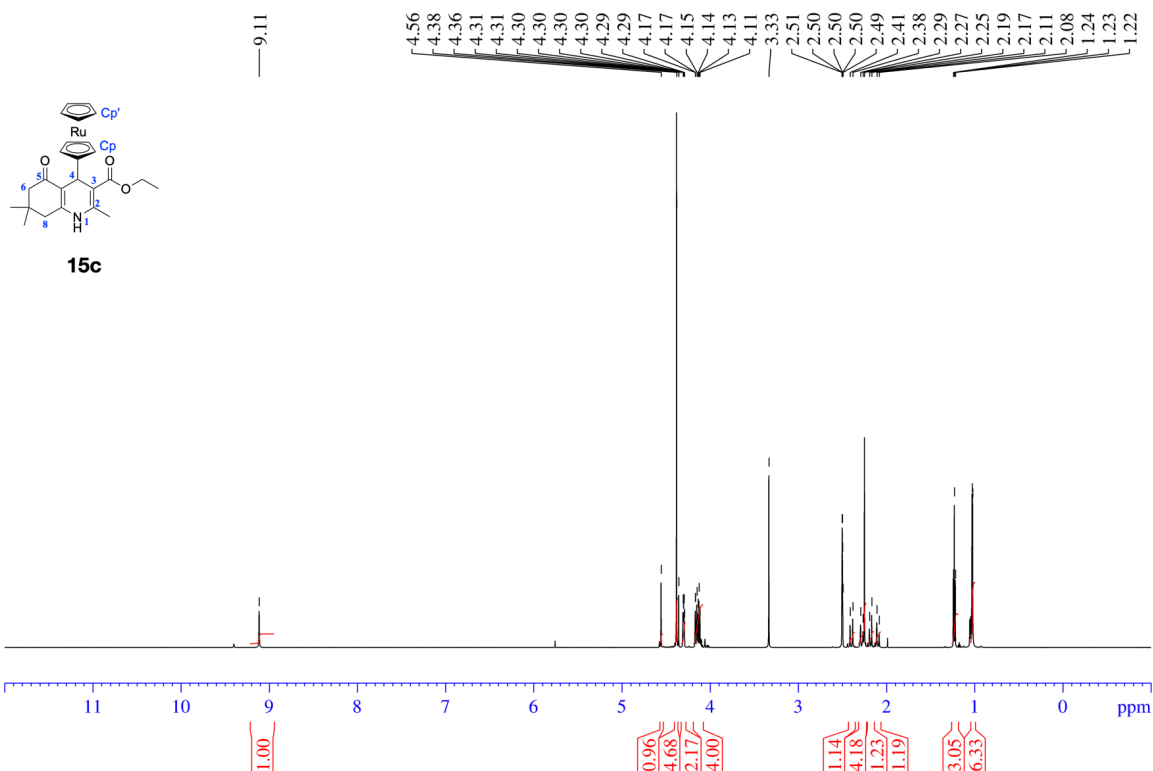
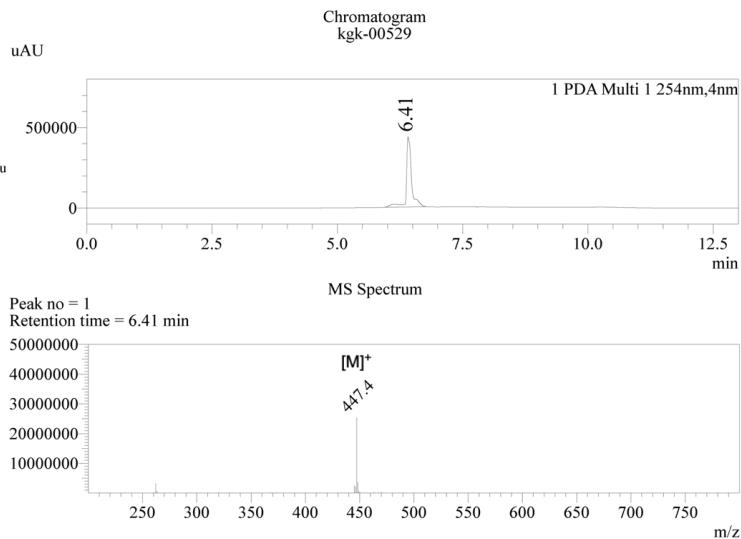


Figure S52. ¹³C{¹H} NMR spectrum of **15c** in DMSO-d₆

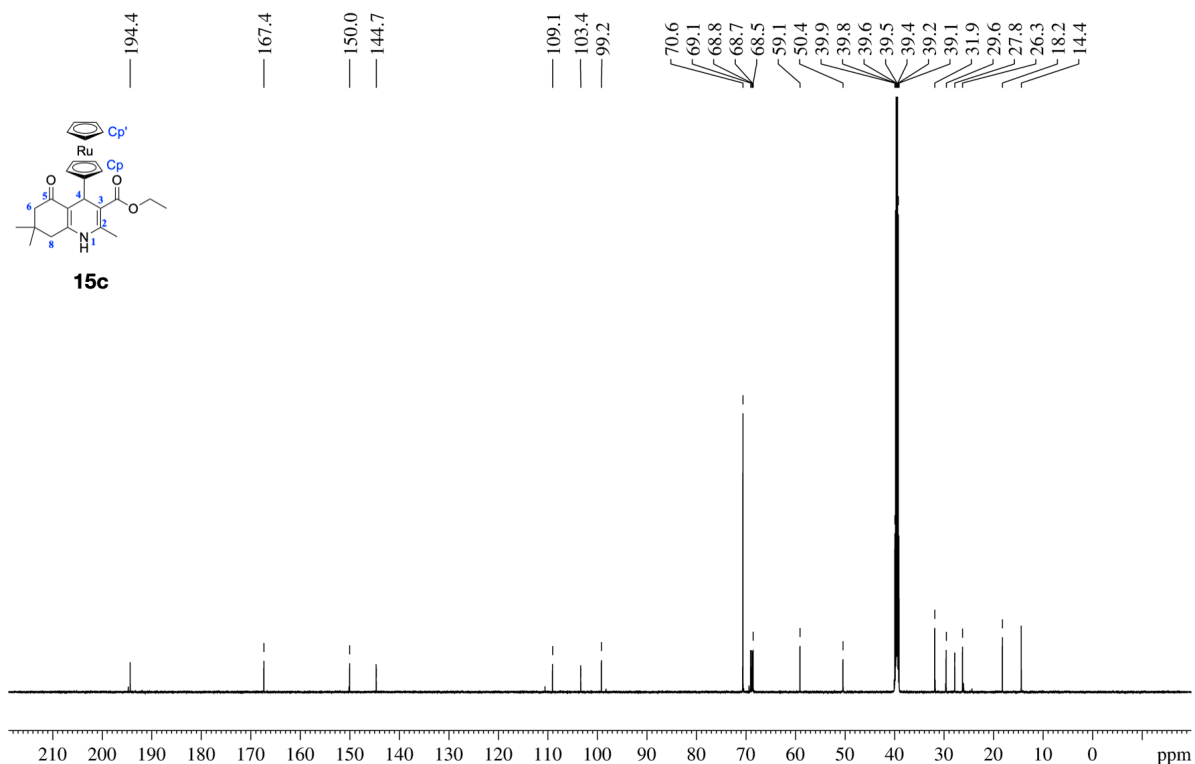


Figure S53. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15c** in DMSO-d_6

Sample Information
 Sample Type : Unknown
 Sample Name : DPX-90032
 Sample ID : DPX-90032
 Vial# : 39
 Injection Volume : 0.3

Method
 <<LC Time Program>>
 Time Module Command
 4.00 Pumps B.Conc
 7.00 Pumps B.Conc
 7.05 Pumps B.Conc
 13.00 Controller Stop

<<Column Information>>
 Column Name : XB-C18
 Column Length : 50 mm
 Inner Diameter : 2.1 mm

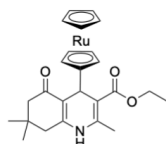
<<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HOAc
 Mobile Phase B : Methanol+0.01%HOAc

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table

Peak#	Ret. Time	Area%
1	6.211	9.644
2	6.356	90.356
Total		100.000



Chemical Formula: $\text{C}_{25}\text{H}_{29}\text{NO}_3\text{Ru}$
 Exact Mass: 493.1

15c

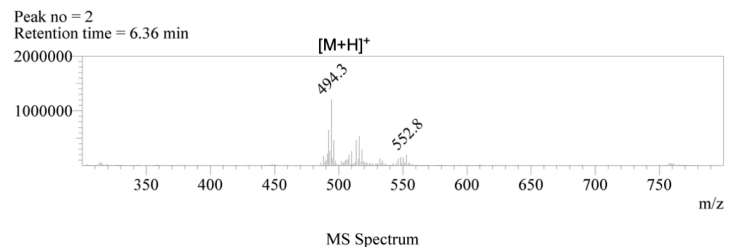
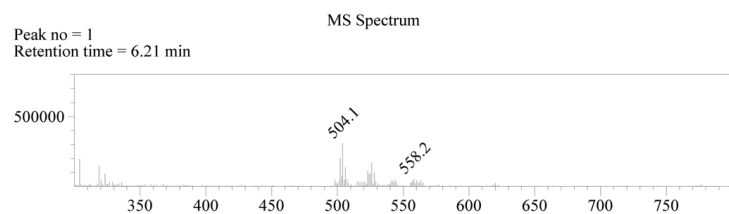
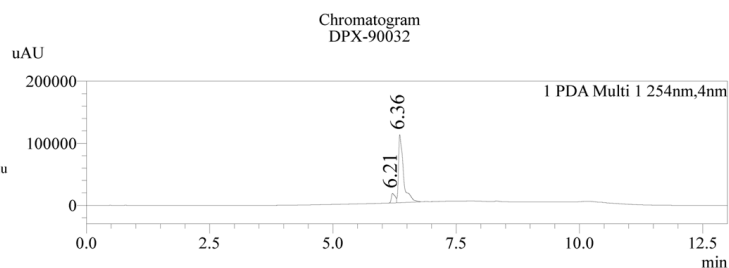


Figure S54. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15c** in DMSO-d_6

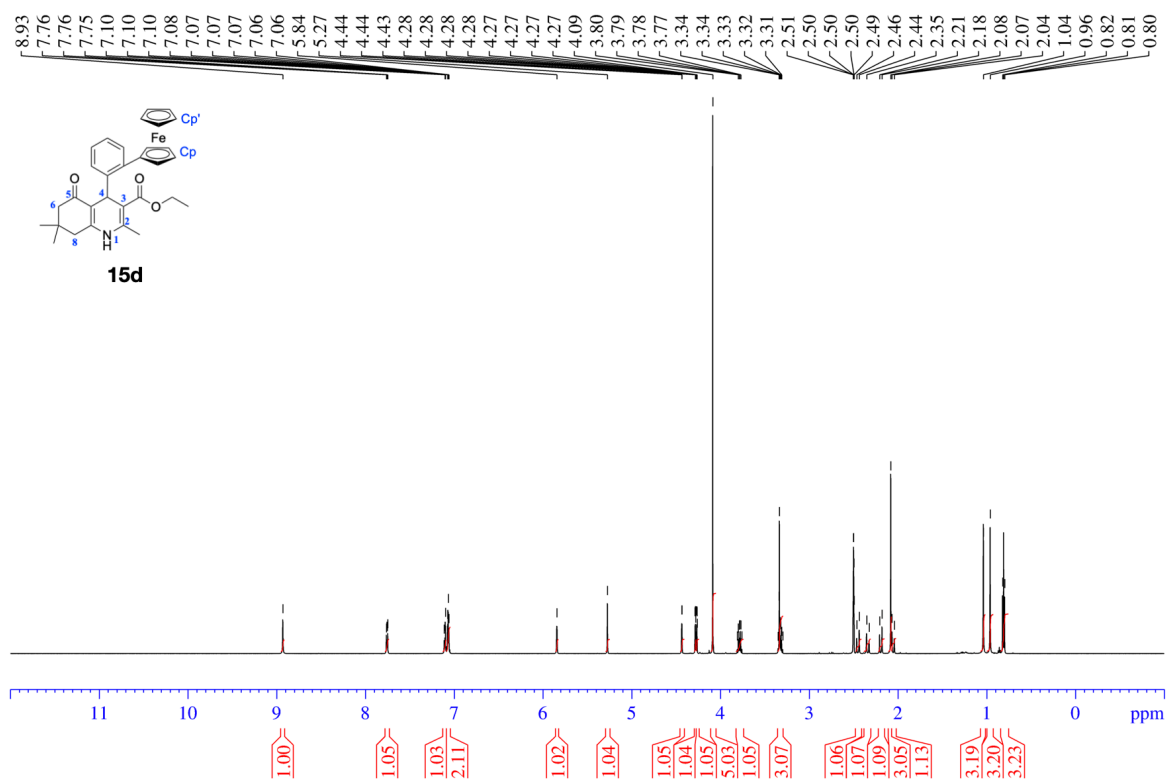


Figure S55. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15d** in $\text{DMSO}-d_6$

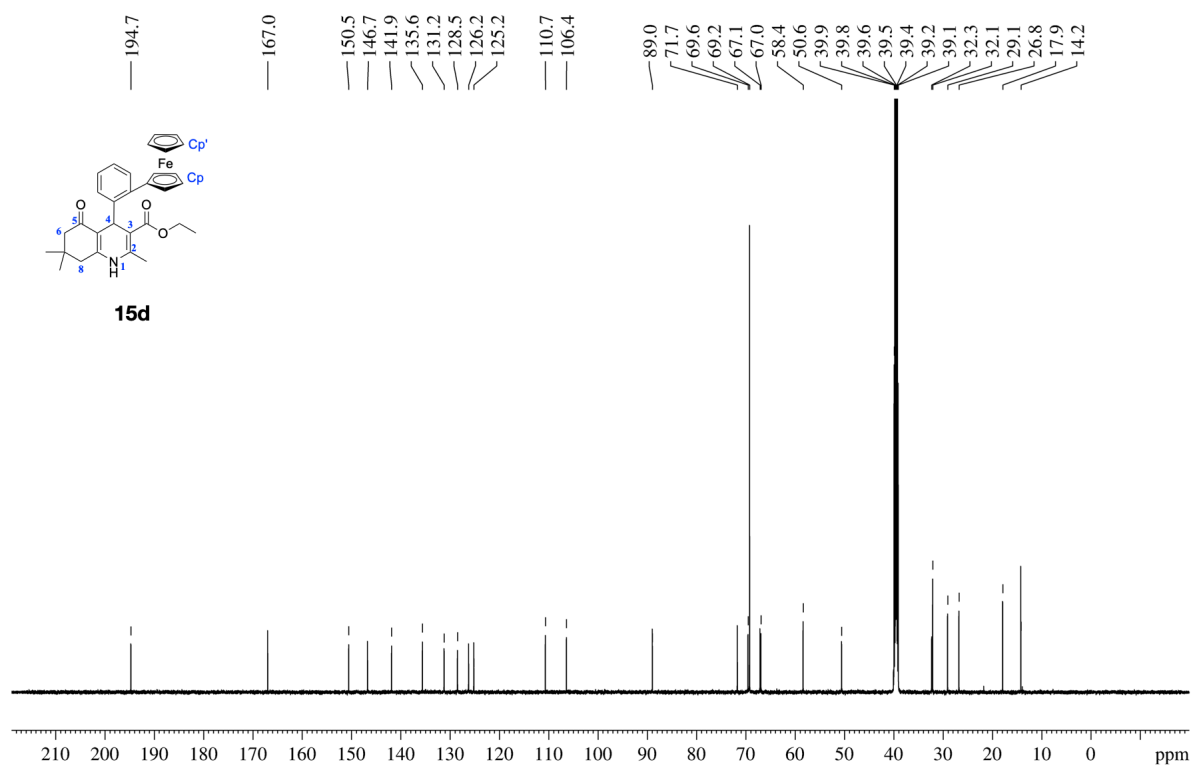


Figure S56. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15d** in $\text{DMSO}-d_6$

Sample Information
 Sample Type : Unknown
 Sample Name : KGK-00528
 Sample ID : KGK-00528
 Vial# : 36
 Injection Volume : 0.3

Method
 <<LC Time Program>>
 Time : 4.00
 Module : Pumps
 7.00 : Pumps
 7.05 : Pumps
 13.00 : Controller
 Command : B.Conc
 B.Conc
 B.Conc
 Stop

<<Column Information>>
 Column Name : XB-C18
 Column Length : 50 mm
 Inner Diameter : 2.1 mm

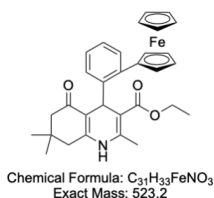
<<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table
 PDA Ch1 254nm

Peak#	Ret. Time	Area%
1	7.072	11.794
2	7.340	88.206
Total		100.000



15d

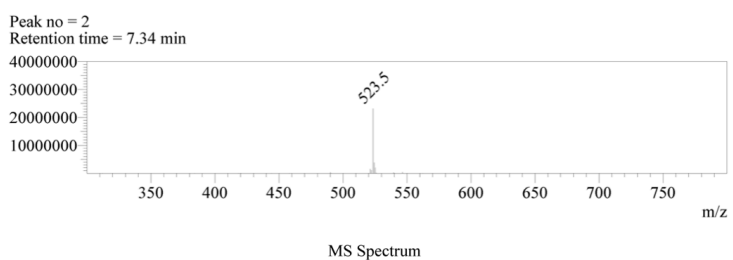
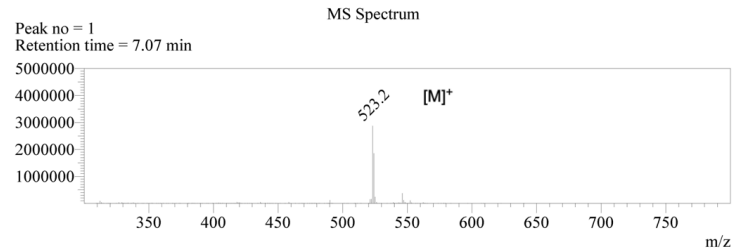
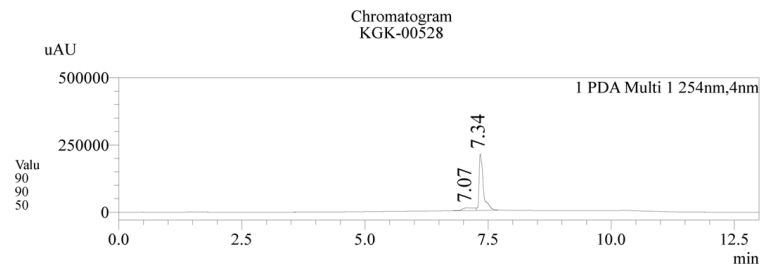


Figure S57. $^{13}C\{^1H\}$ NMR spectrum of **15d** in $DMSO-d_6$

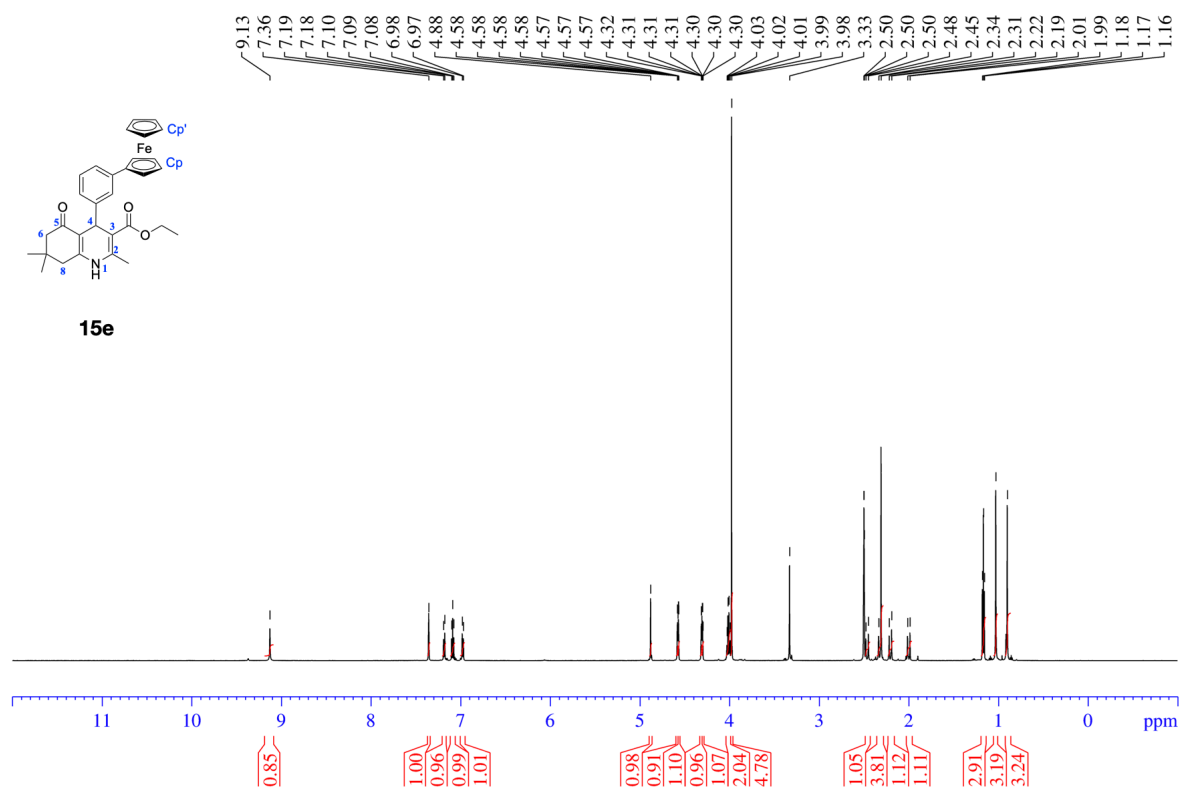


Figure S58. $^{13}C\{^1H\}$ NMR spectrum of **15e** in $DMSO-d_6$

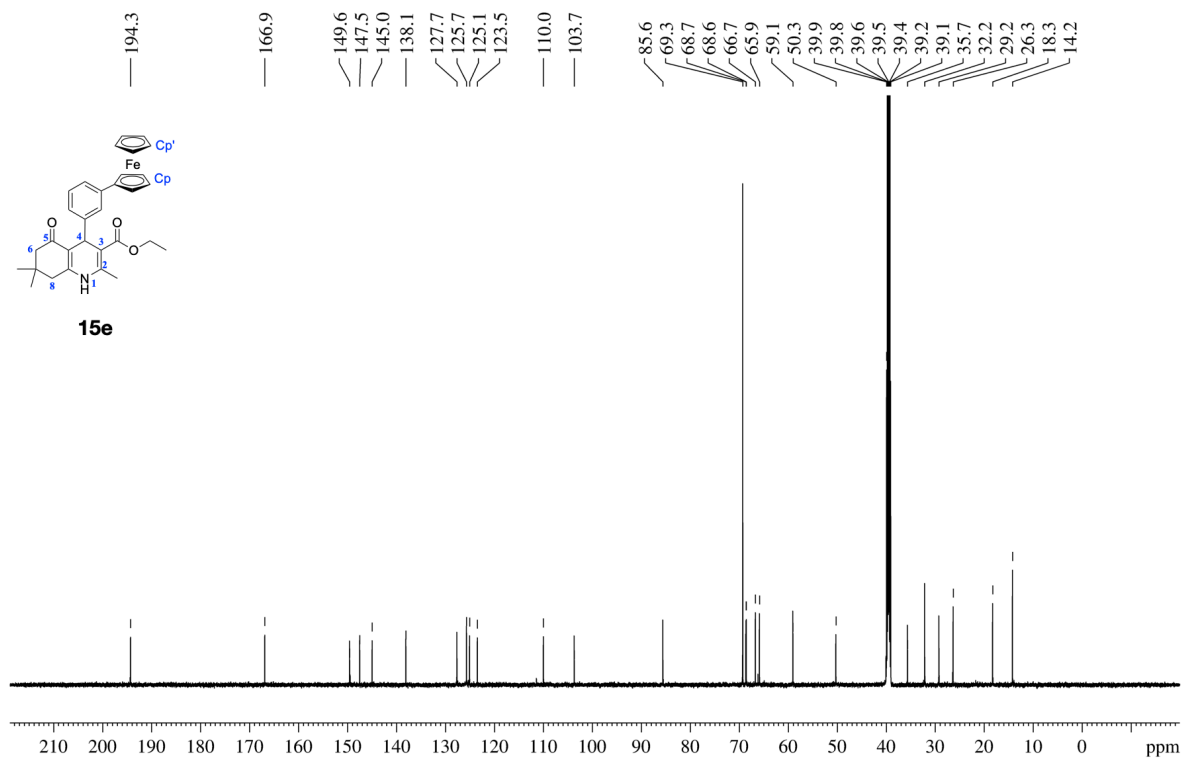


Figure S59. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15e** in DMSO-d_6

Sample Information
 Sample Type : Unknown
 Sample Name : DPX-90007
 Sample ID : DPX-90007
 Vial# : 45
 Injection Volume : 0.3

Method
 Time : 4.00
 Module : Pumps
 Pumps : B.Conc
 7.00 : Pumps
 B.Conc
 7.05 : Pumps
 B.Conc
 13.00 : Controller
 Stop

<<Column Information>>
 Column Name : XB-C18
 Column Length : 50 mm
 Inner Diameter : 2.1 mm

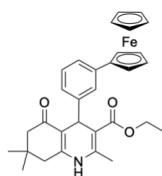
<<Mobile Phase Name>>
 Mobile Phase A : water+0.01% HCOOH
 Mobile Phase B : Methanol+0.01% HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

PDA Ch1 254nm

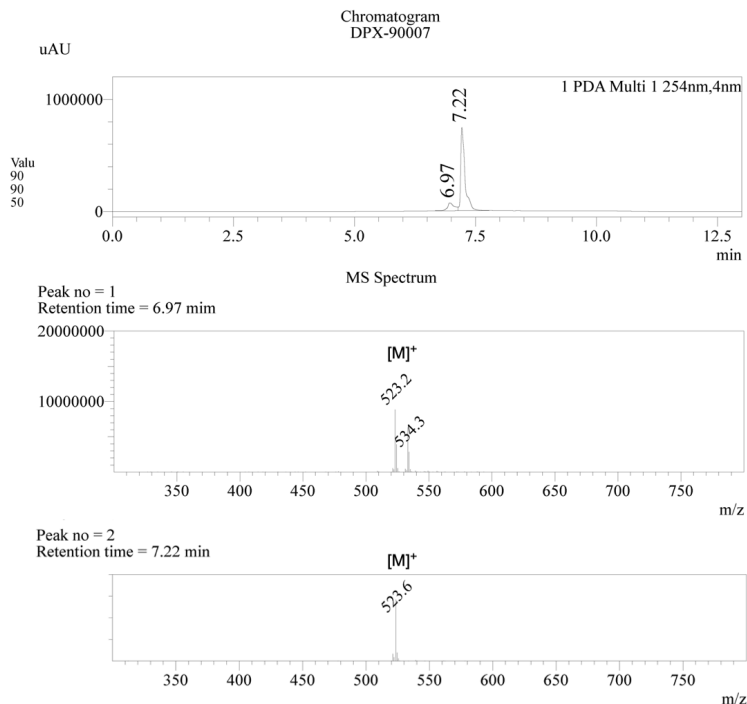
Peak#	Ret. Time	Area%
1	6.968	13.836
2	7.219	86.164
Total		100.000



Chemical Formula: $\text{C}_{31}\text{H}_{33}\text{FeNO}_3$
 Exact Mass: 523.2

15e

Figure S60. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15e** in DMSO-d_6



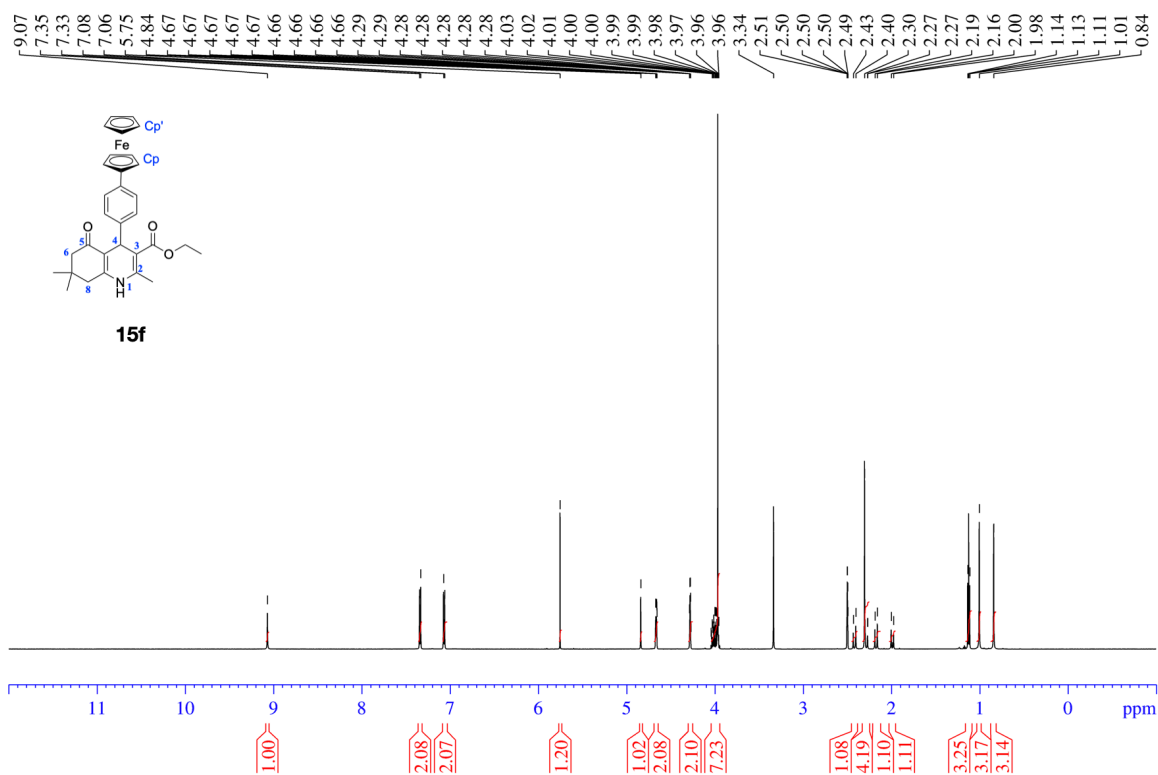


Figure S61. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15f** in DMSO-d_6

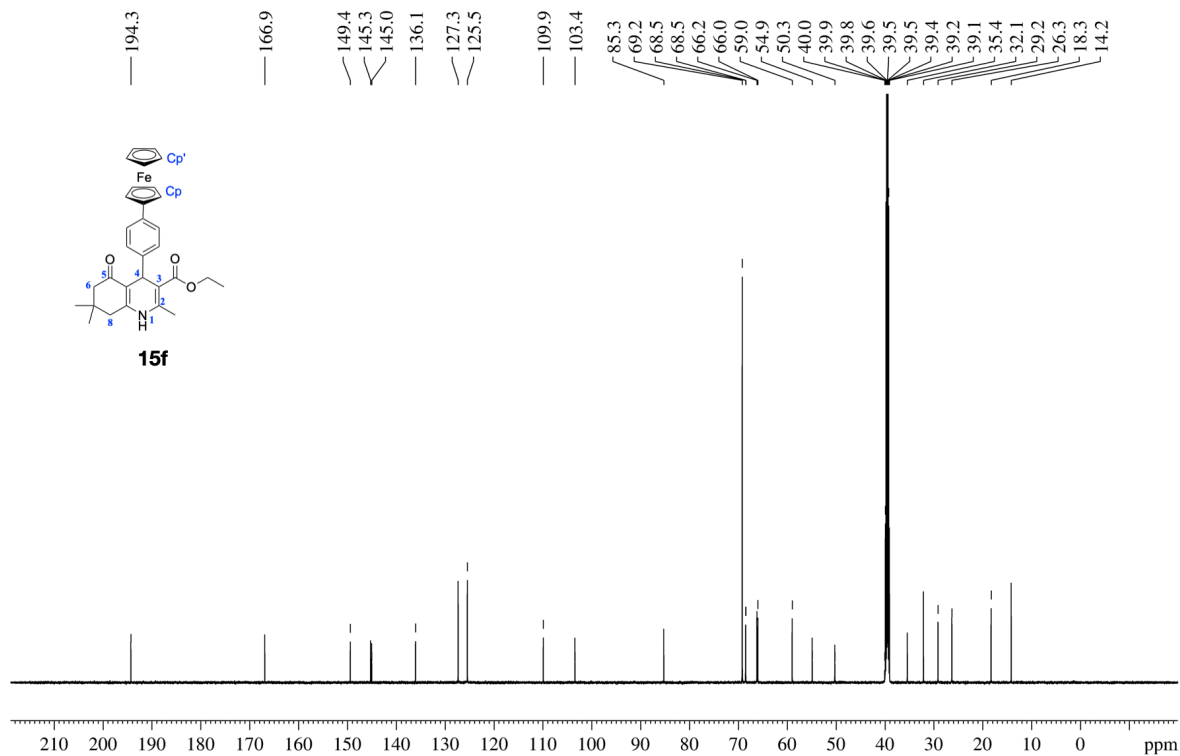


Figure S62. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15f** in DMSO-d_6

Sample Information
 Sample Type : Unknown
 Sample Name : kgk-00527_MeOH_5-9-5
 Sample ID : kgk-00527_MeOH_5-9-5
 Vial# : 97
 Injection Volume : 0.5

Method
 <<LC Time Program>>
 Time Module Command Valu
 4.00 Pumps B.Conc 90
 7.00 Pumps B.Conc 90
 7.05 Pumps B.Conc 50
 13.00 Controller Stop

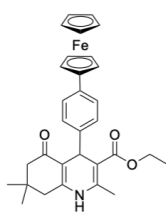
<<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table

Peak#	Ret. Time	Area%
1	8.002	4.595
2	8.205	95.405
Total		100.000



Chemical Formula: C₃₁H₃₃FeNO₃
 Exact Mass: 523,2

15f

Figure S63. ¹³C{¹H} NMR spectrum of 15f in DMSO-d₆

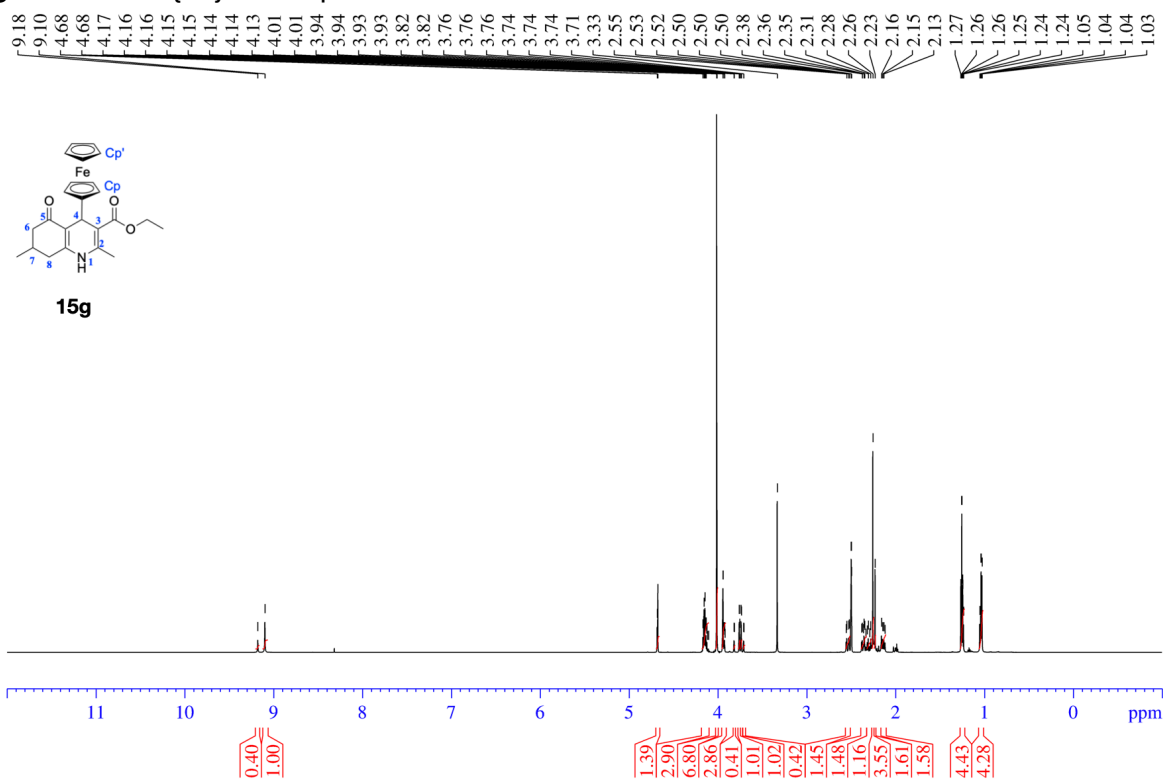
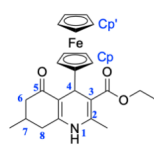


Figure S64. ¹³C{¹H} NMR spectrum of 15g in DMSO-d₆



15g

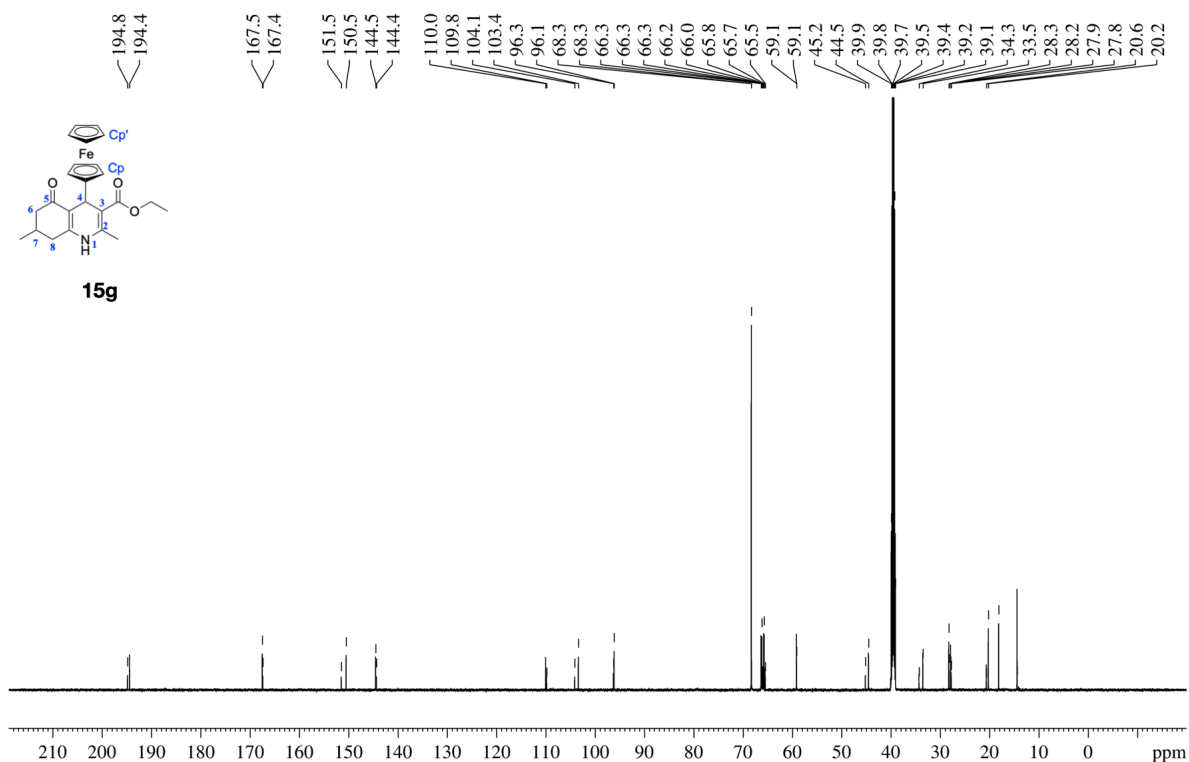


Figure S65. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15g** in DMSO-d_6

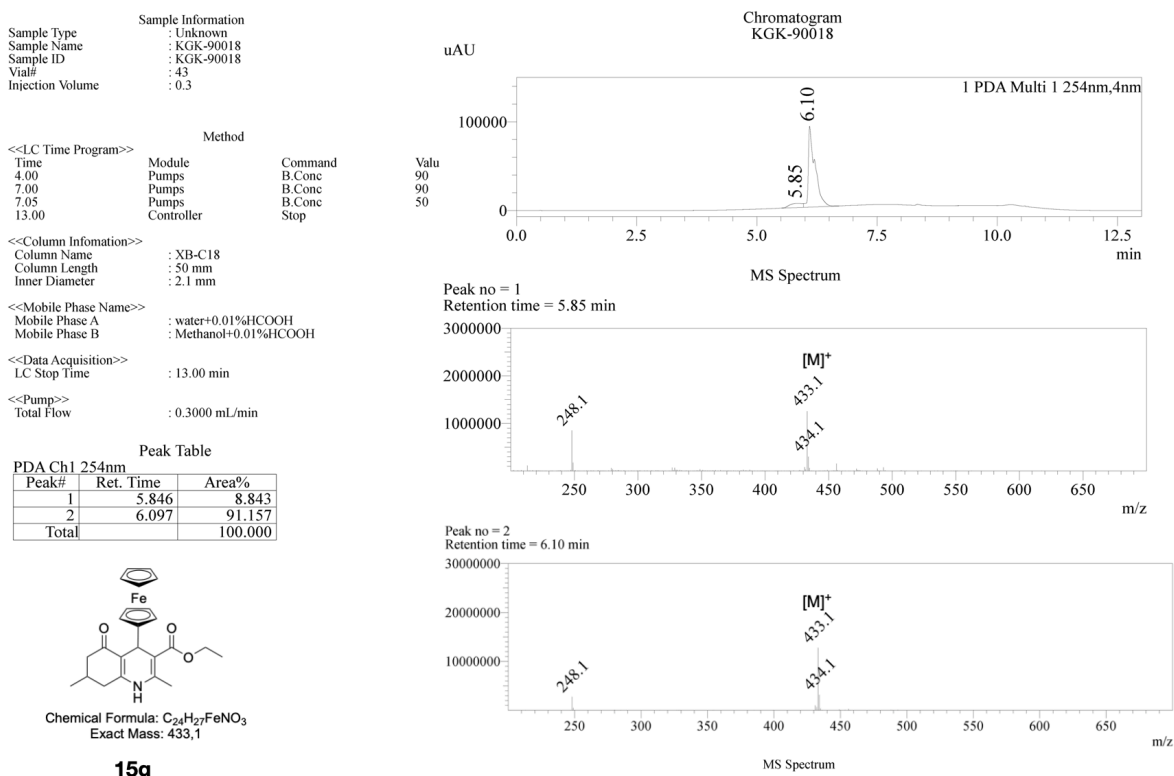


Figure S66. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15g** in DMSO-d_6

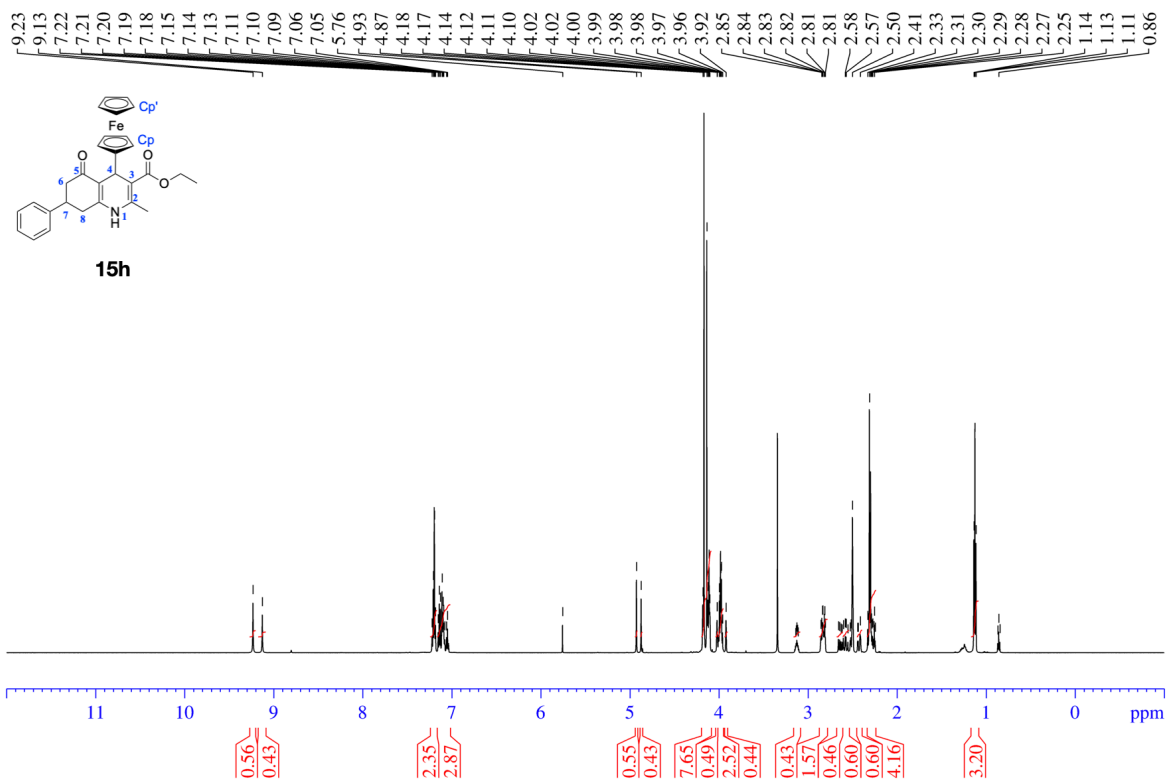


Figure S67. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15h** in DMSO-d_6

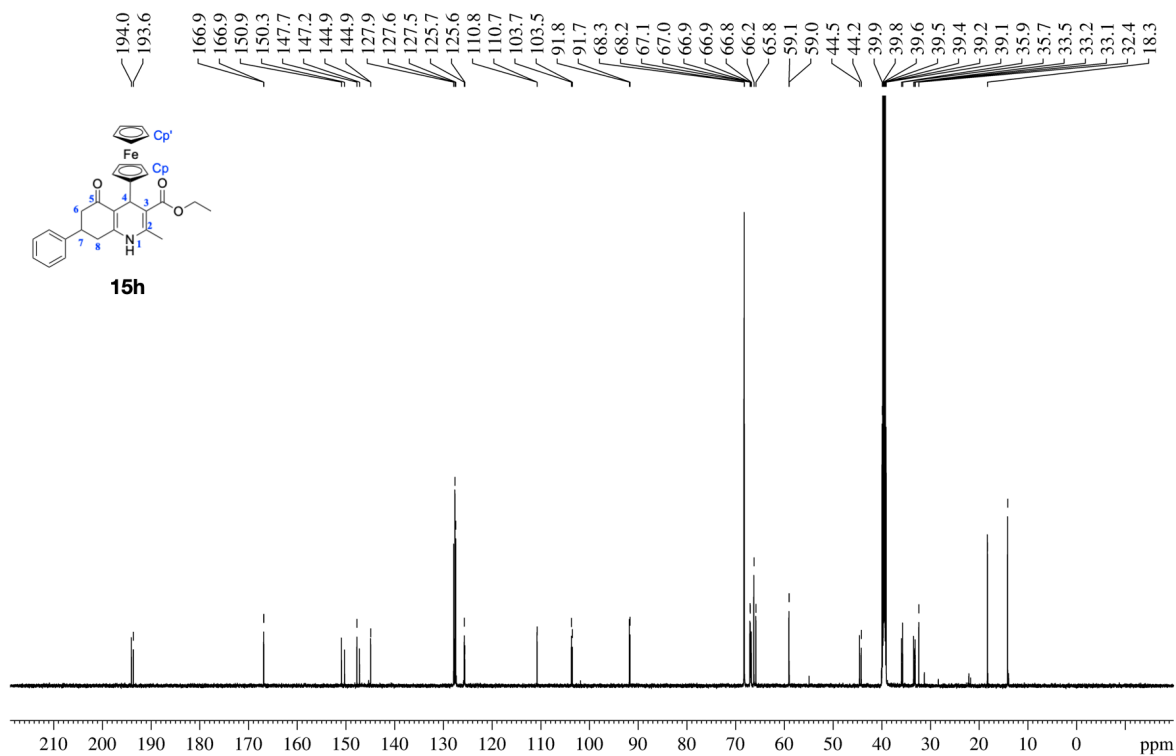


Figure S68. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15h** in DMSO-d_6

Sample Information
 Sample Type : Unknown
 Sample Name : KGK-90013
 Sample ID : KGK-90013
 Vial#: 38
 Injection Volume : 0.3

Method
 Module : Pumps
 Command : B.Conc
 Pumps : B.Conc
 Controller : B.Conc
 Stop

<<LC Time Program>>
 Time : 4.00
 7.00
 7.05
 13.00

<<Column Information>>
 Column Name : XB-C18
 Column Length : 50 mm
 Inner Diameter : 2.1 mm

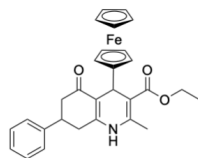
<<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table

Peak#	Ret. Time	Area%
1	5.752	1.483
2	6.786	49.413
3	6.942	49.104
Total		100.000



Chemical Formula: C₂₉H₂₉FeNO₃
 Exact Mass: 495.1

15h

Figure S69. ¹³C{¹H} NMR spectrum of 15h in DMSO-d₆

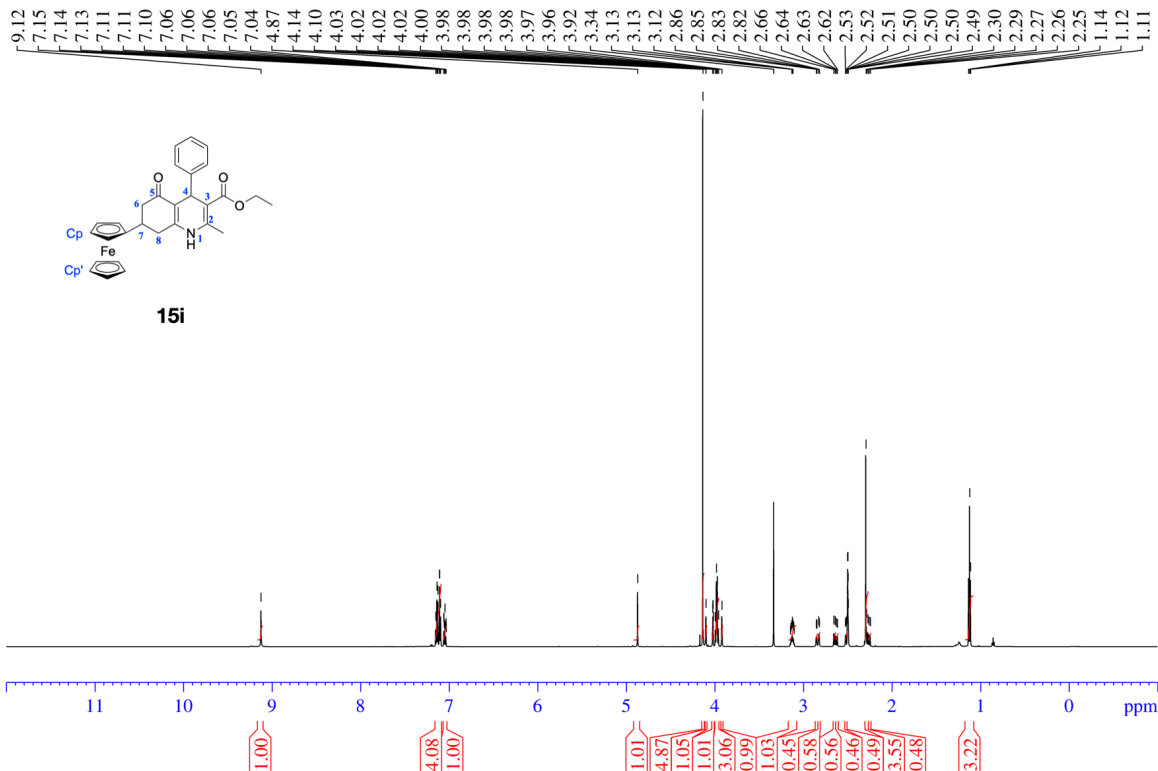
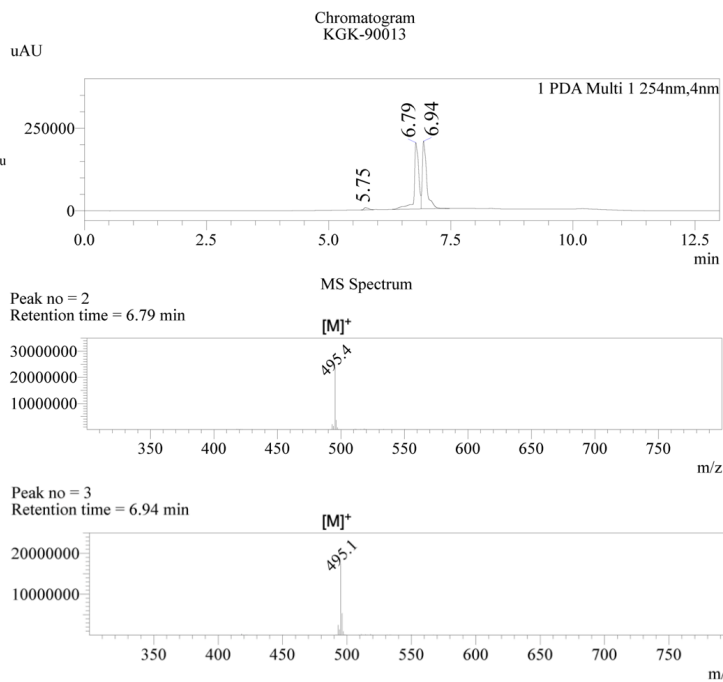


Figure S70. ¹³C{¹H} NMR spectrum of 15i in DMSO-d₆



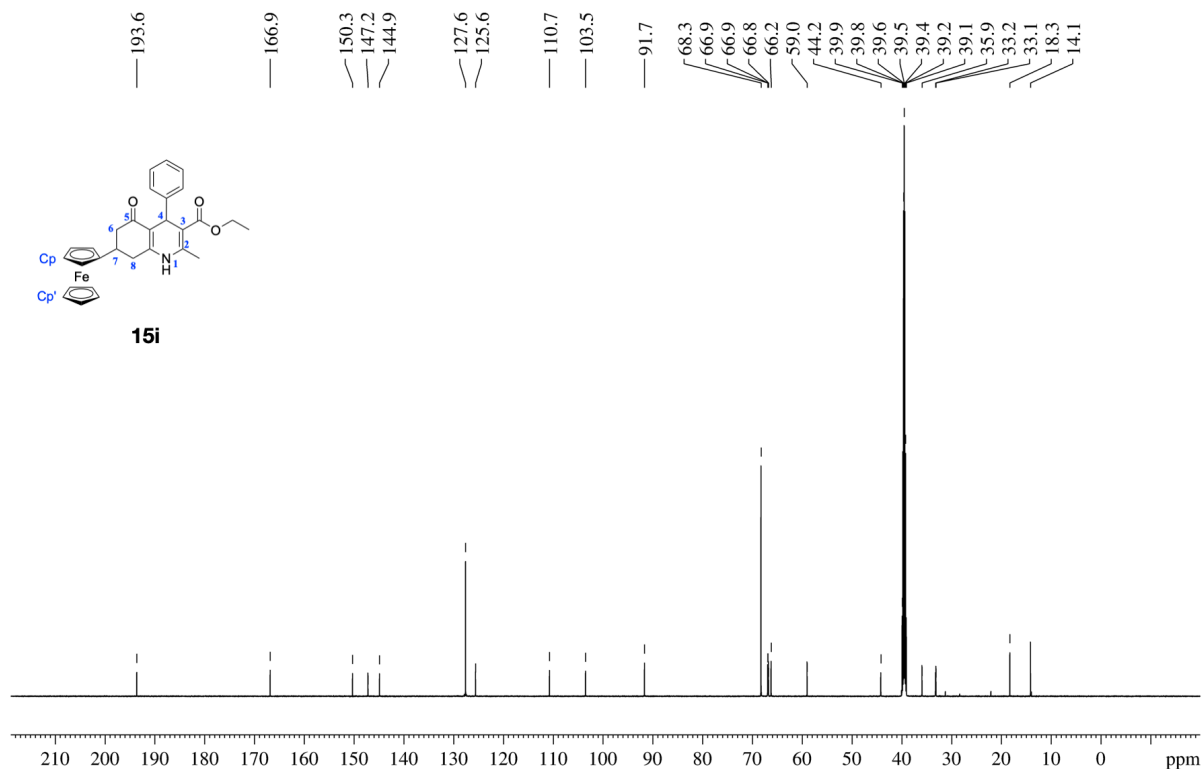


Figure S71. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15i** in $\text{DMSO}-d_6$

Sample Information
 Sample Type : Unknown
 Sample Name : kgk-00545_MeOH_5-9-5
 Sample ID : kgk-00545_MeOH_5-9-5
 Vial# : 101
 Injection Volume : 0.5

Method
 Module Command
 Pumps B.Conc
 Pumps B.Conc
 Pumps B.Conc
 Controller Stop

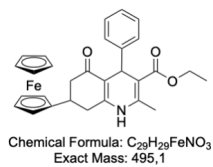
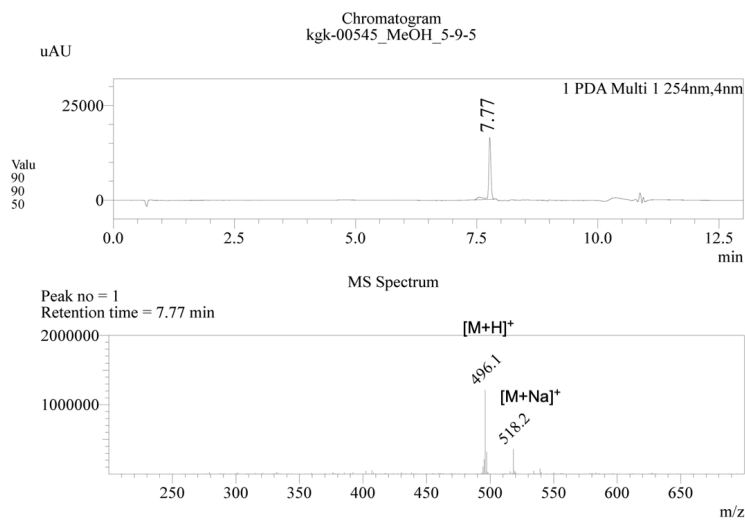
<<LC Time Program>>
 Time 4.00
 7.00
 7.05
 13.00

<<Mobile Phase Name>>
 Mobile Phase A : water+0.01% HCOOH
 Mobile Phase B : Methanol+0.01% HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table		
Peak#	Ret. Time	Area%
1	7.770	100.000
Total		100.000



15i

Figure S72. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15i** in $\text{DMSO}-d_6$

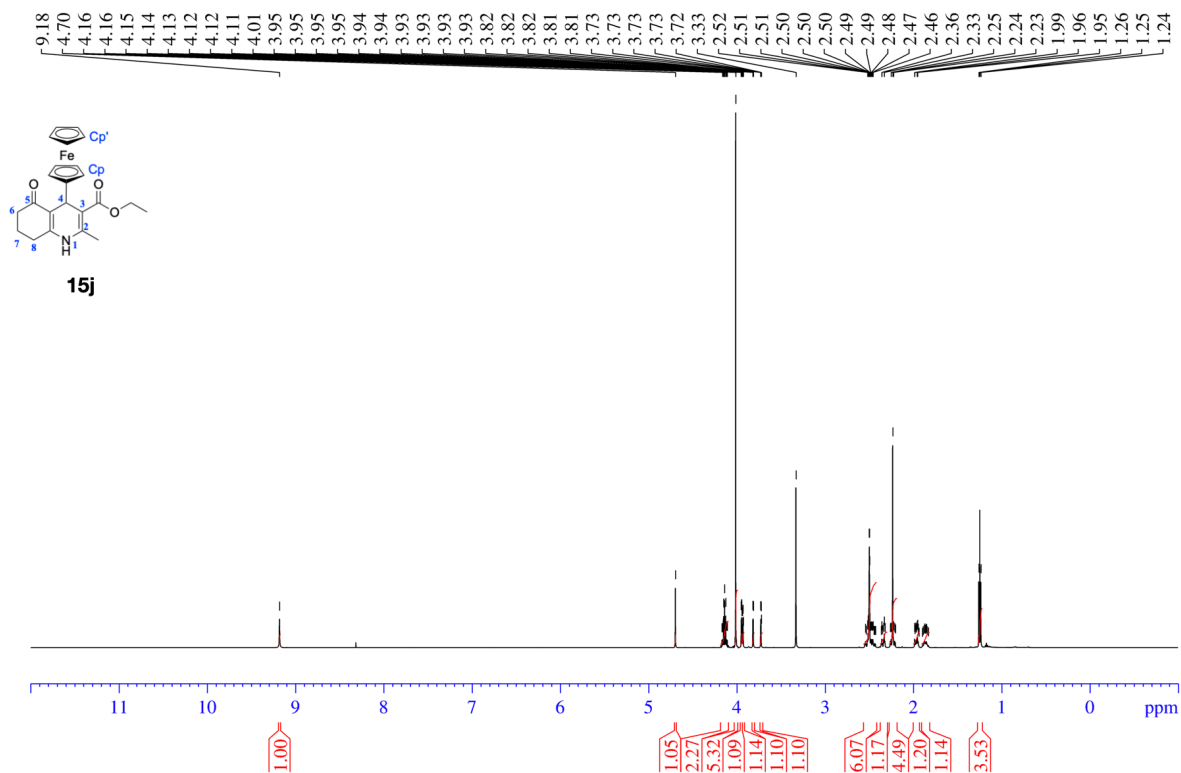


Figure S73. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15i** in DMSO-d_6

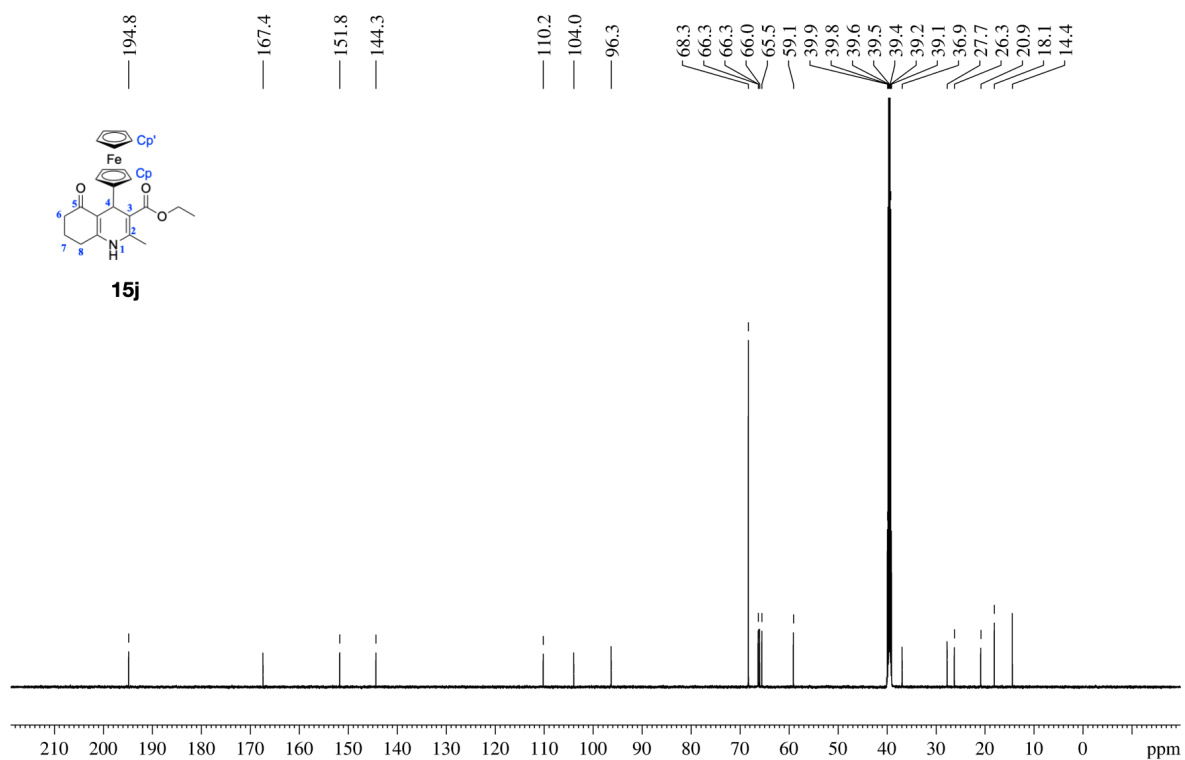


Figure S74. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **15j** in DMSO-d_6

Sample Information
 Sample Type : Unknown
 Sample Name : KKG-90005
 Sample ID : KKG-90005
 Vial# : 71
 Injection Volume : 2

Method
 <<LC Time Program>>
 Time Module Command
 4.00 Pumps B.Conc 90
 7.00 Pumps B.Conc 90
 7.00 Pumps B.Conc 90
 13.00 Controller Stop

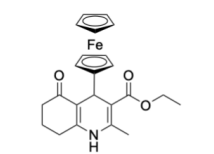
<<Mobile Phase Name>>
 Mobile Phase A : water+0.01%*HCOOH*
 Mobile Phase B : Methanol+0.01%*HCOOH*

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table

Peak#	Ret. Time	Area%
1	7.032	11.735
2	7.369	88.265
Total		100.000



15j

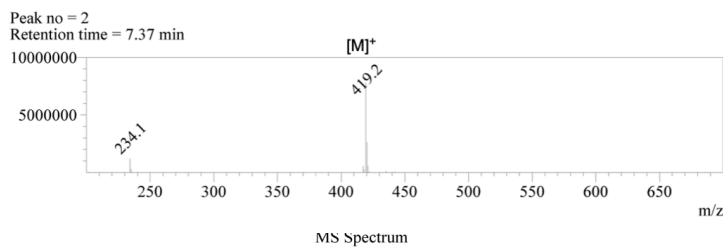
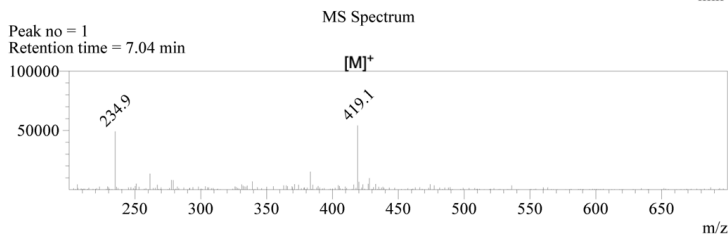
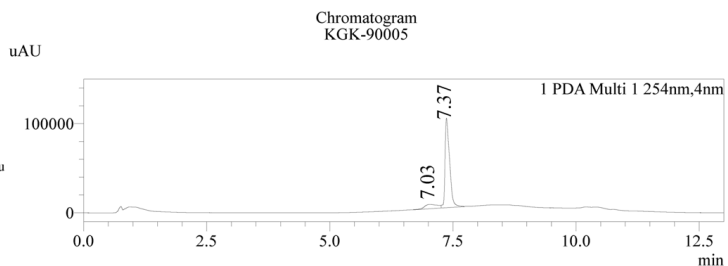


Figure S75. $^{13}C\{^1H\}$ NMR spectrum of 15j in $DMSO-d_6$

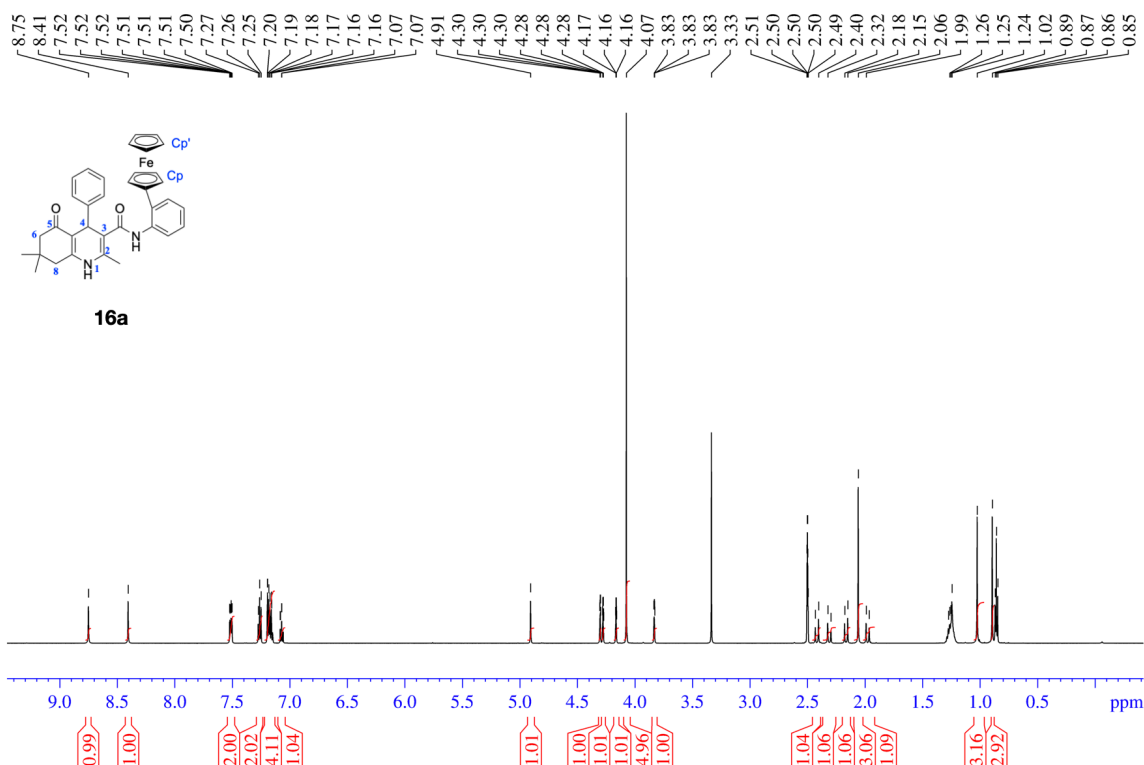


Figure S76. $^{13}C\{^1H\}$ NMR spectrum of 16a in $DMSO-d_6$

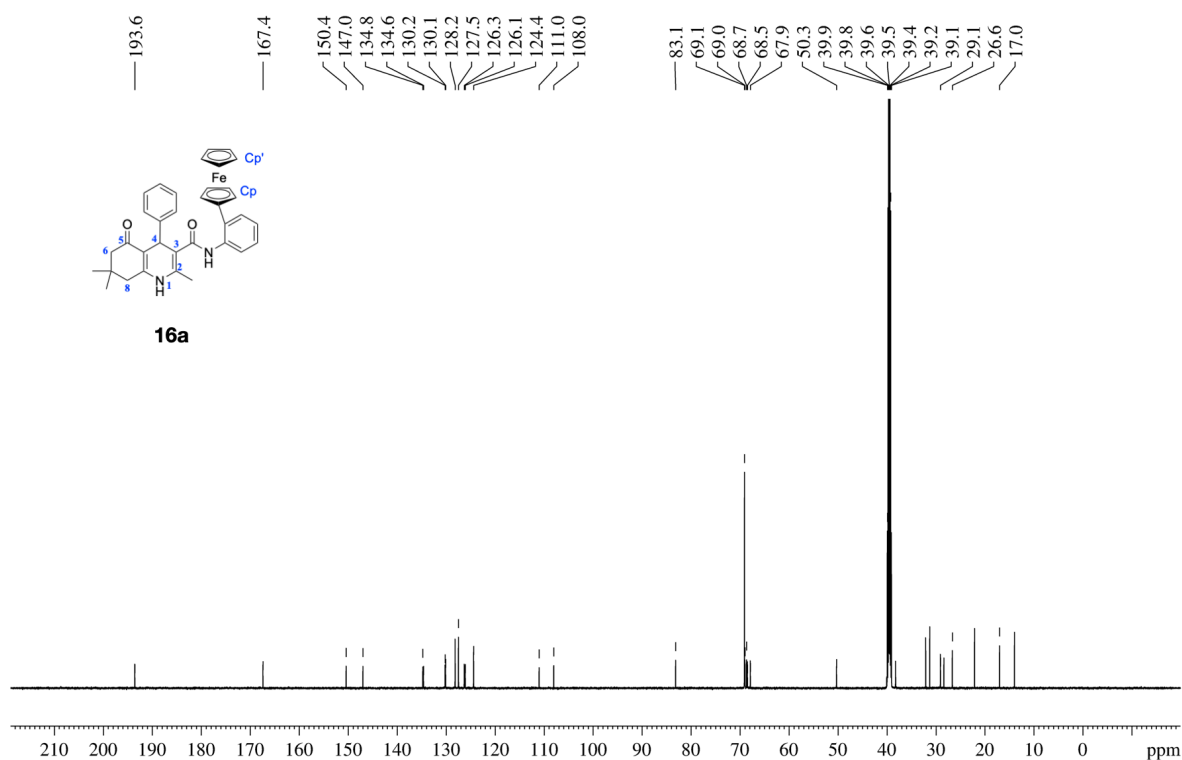


Figure S77. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **16a** in $\text{DMSO-}d_6$

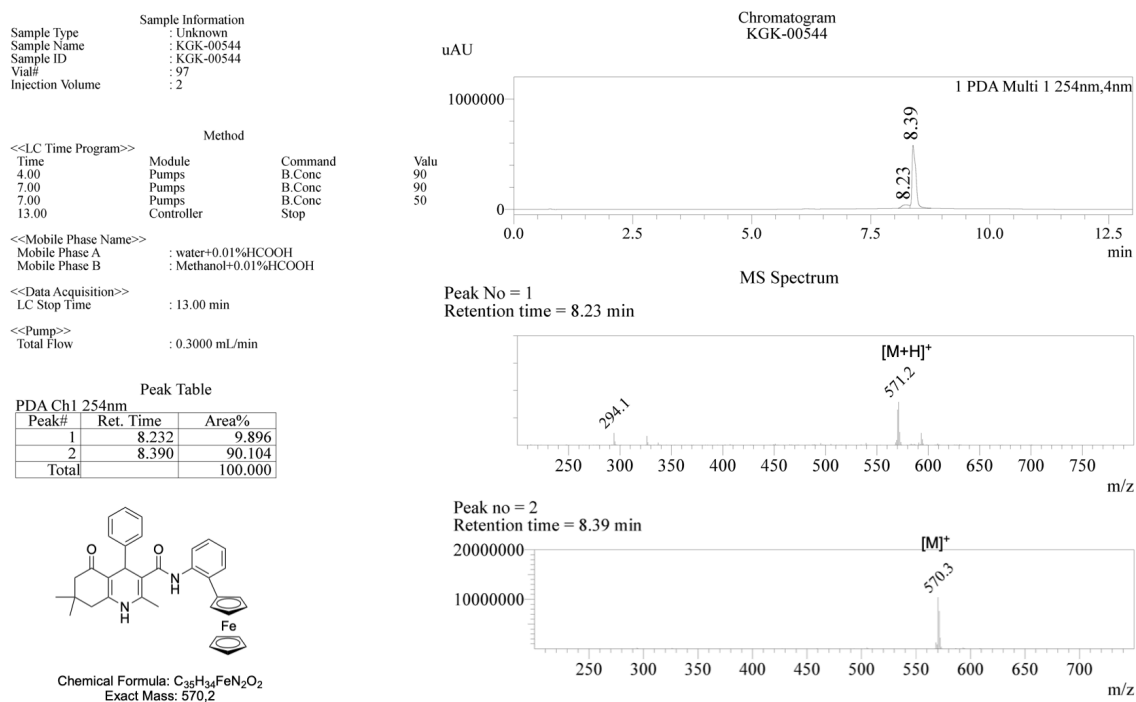


Figure S78. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **16a** in $\text{DMSO-}d_6$

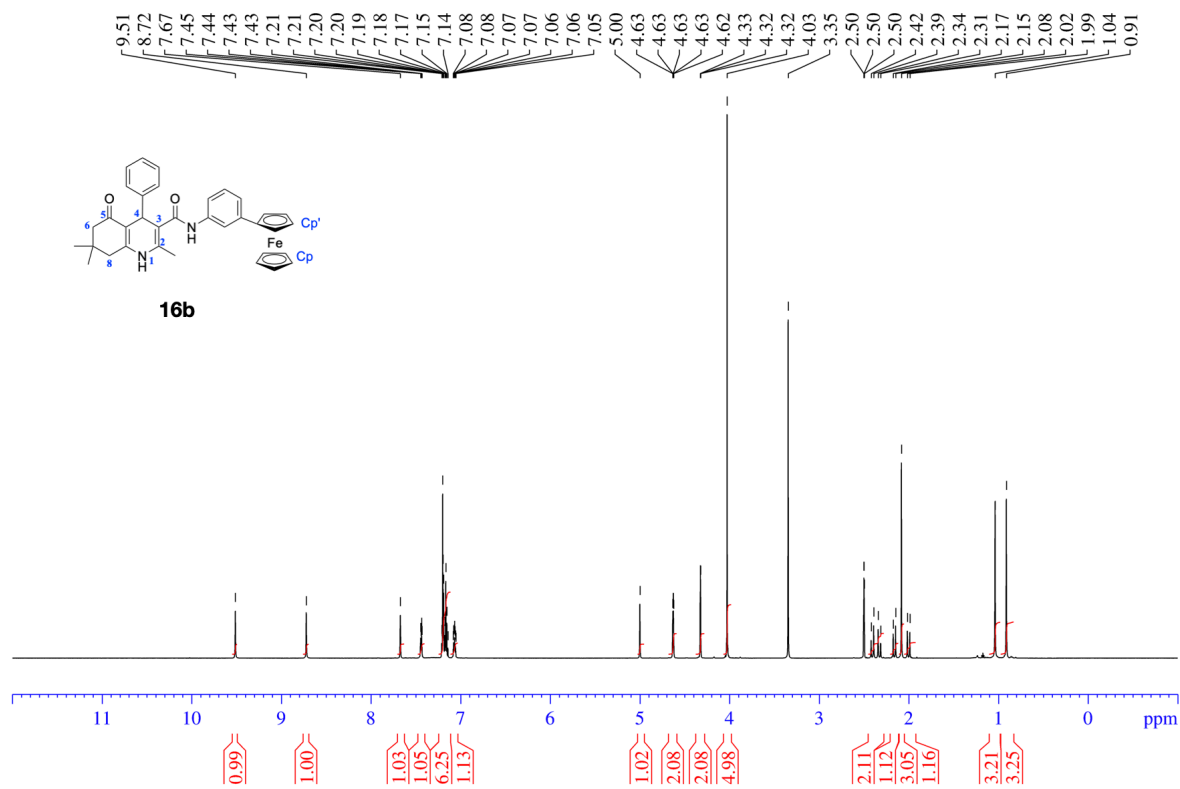


Figure S79. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **16b** in DMSO-d_6

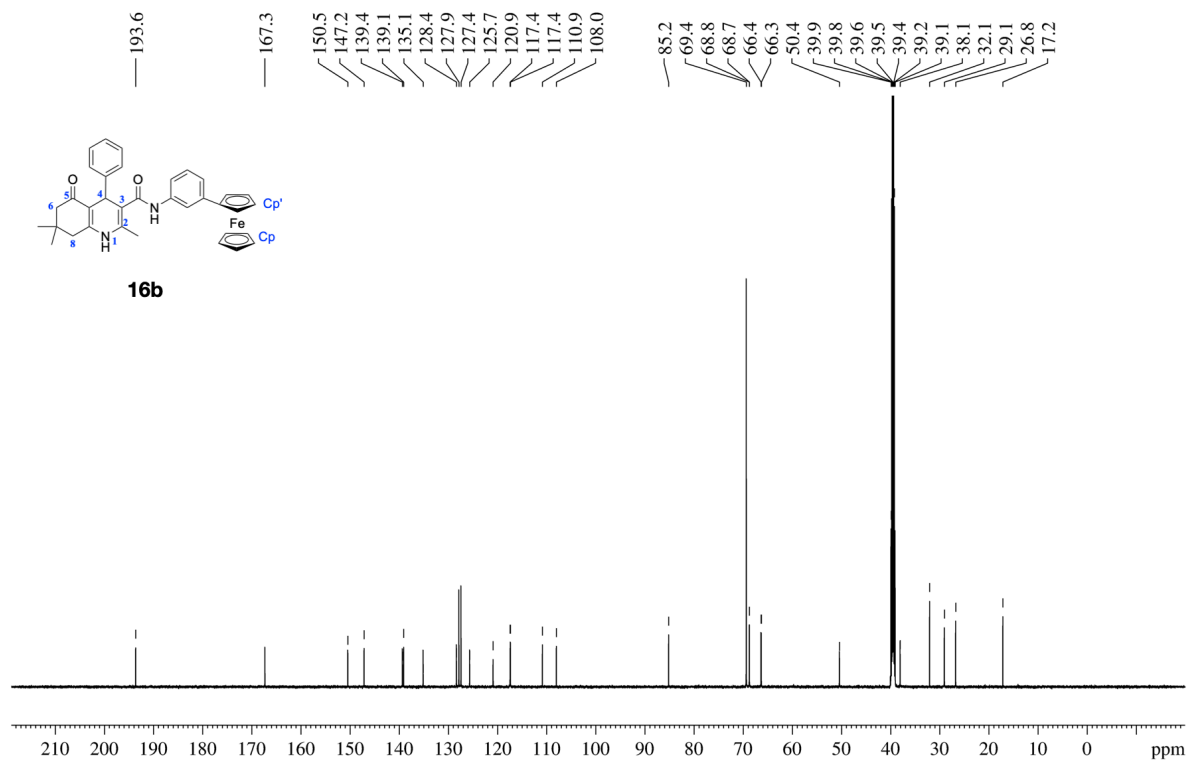


Figure S80. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **16b** in DMSO-d_6

Sample Information
 Sample Type : Unknown
 Sample Name : KGK-00540
 Sample ID : KGK-00540
 Vial# : 96
 Injection Volume : 2

Method
 <<LC Time Program>>
 Time Module Command Valu
 4.00 Pumps B.Conc 90
 7.00 Pumps B.Conc 90
 7.00 Pumps B.Conc 50
 13.00 Controller Stop

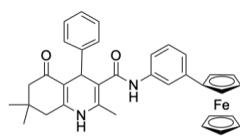
<<Mobile Phase Name>>
 Mobile Phase A : water+0.01%HCOOH
 Mobile Phase B : Methanol+0.01%HCOOH

<<Data Acquisition>>
 LC Stop Time : 13.00 min

<<Pump>>
 Total Flow : 0.3000 mL/min

Peak Table

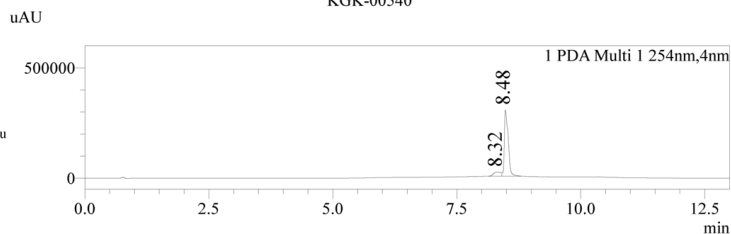
Peak#	Ret. Time	Area%
1	8.319	10.734
2	8.481	89.266
Total		100.000



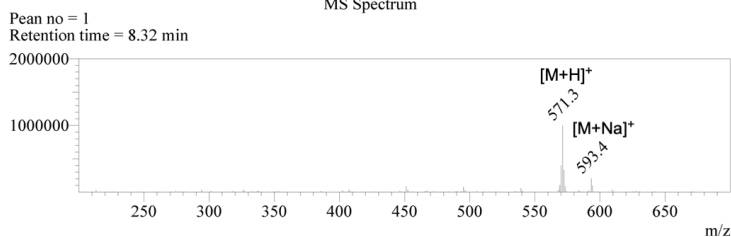
Chemical Formula: C₃₅H₃₄FeN₂O₂
 Exact Mass: 570.2

16b

Chromatogram
 KGK-00540



MS Spectrum



Peak no = 2
 Retention time = 8.48 min

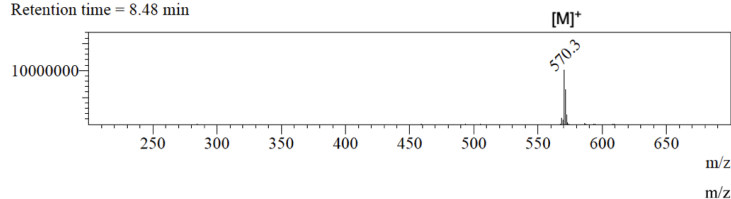


Figure S81. ¹³C{¹H} NMR spectrum of 16b in DMSO-d₆

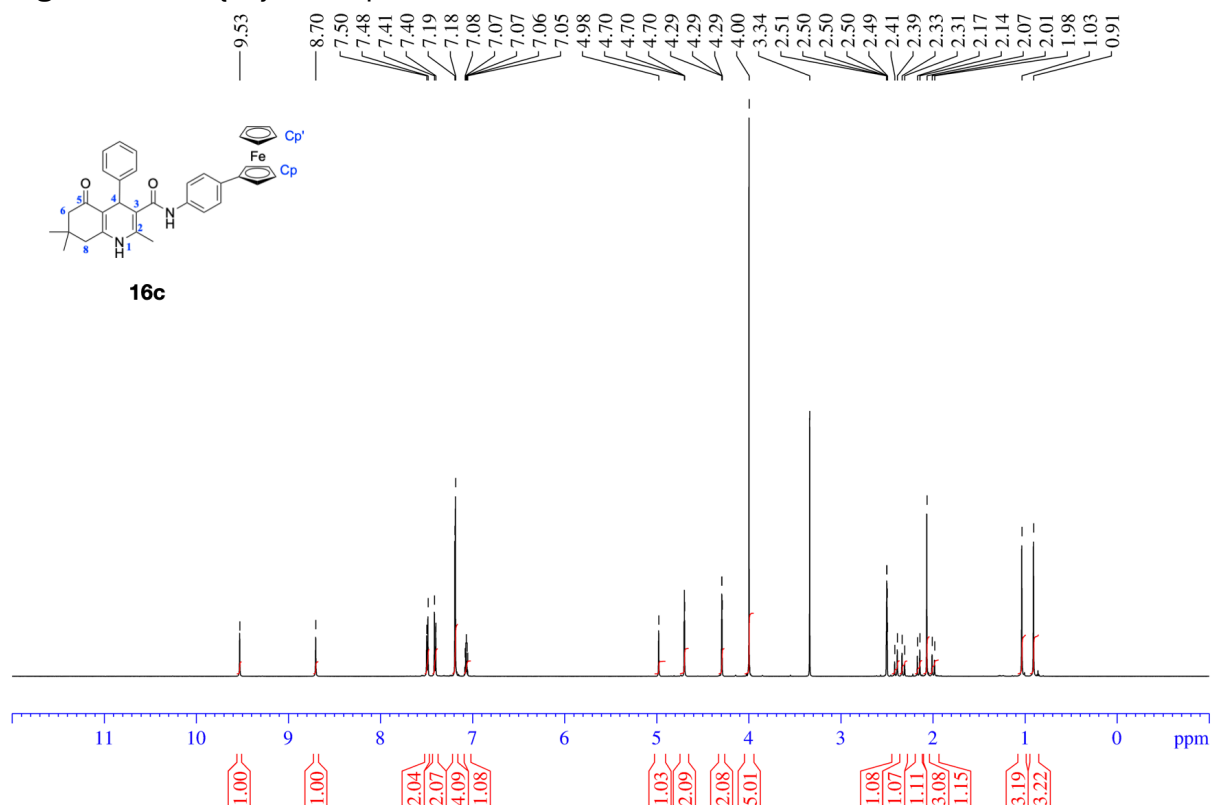
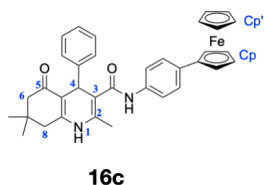


Figure S82. ¹³C{¹H} NMR spectrum of 16c in DMSO-d₆



16c

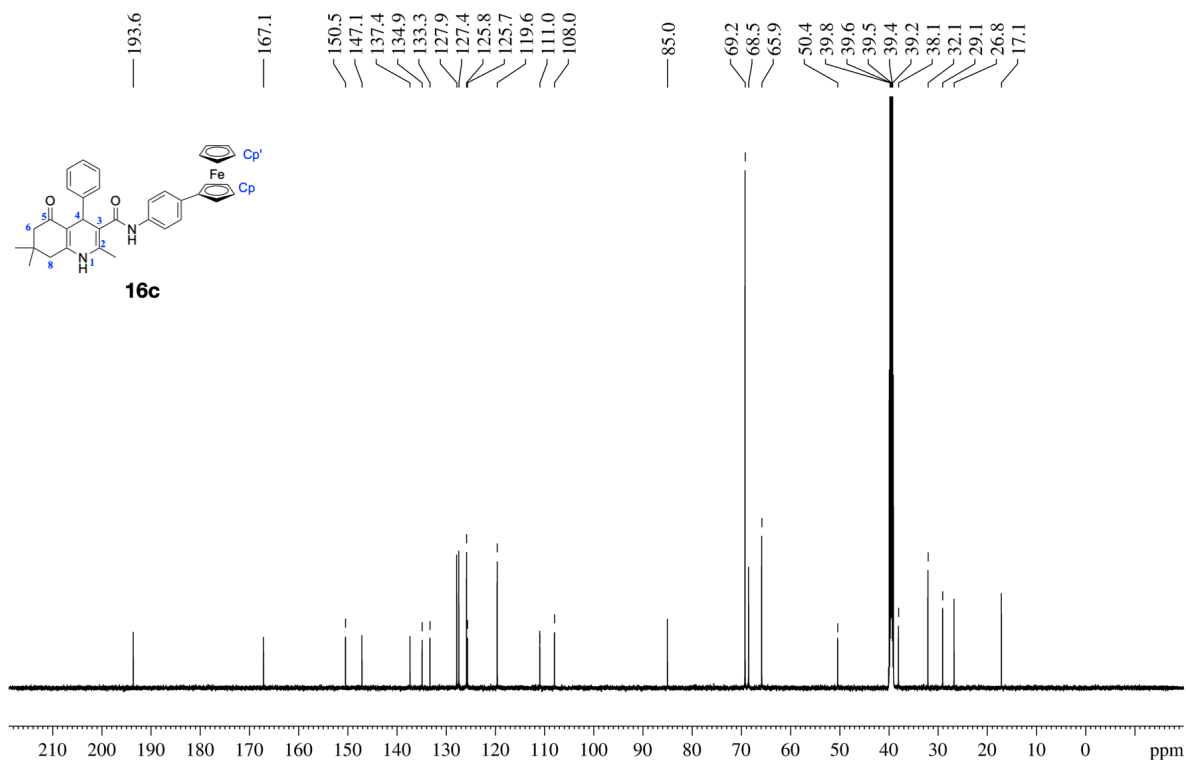


Figure S83. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **16c** in DMSO-d_6

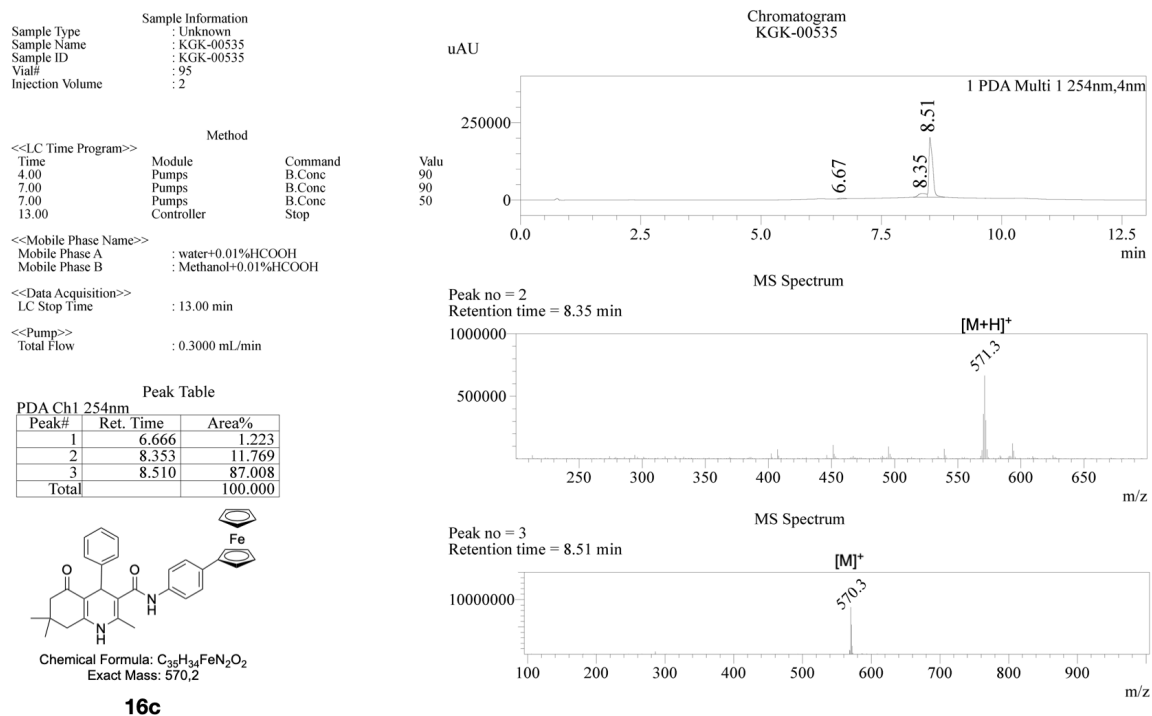


Figure S84. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **16c** in DMSO-d_6