Electronic Supporting Information

Antitumor and biological investigation of doubly cyclometalated ruthenium(II) organometallics derived from benzimidazolyl derivatives

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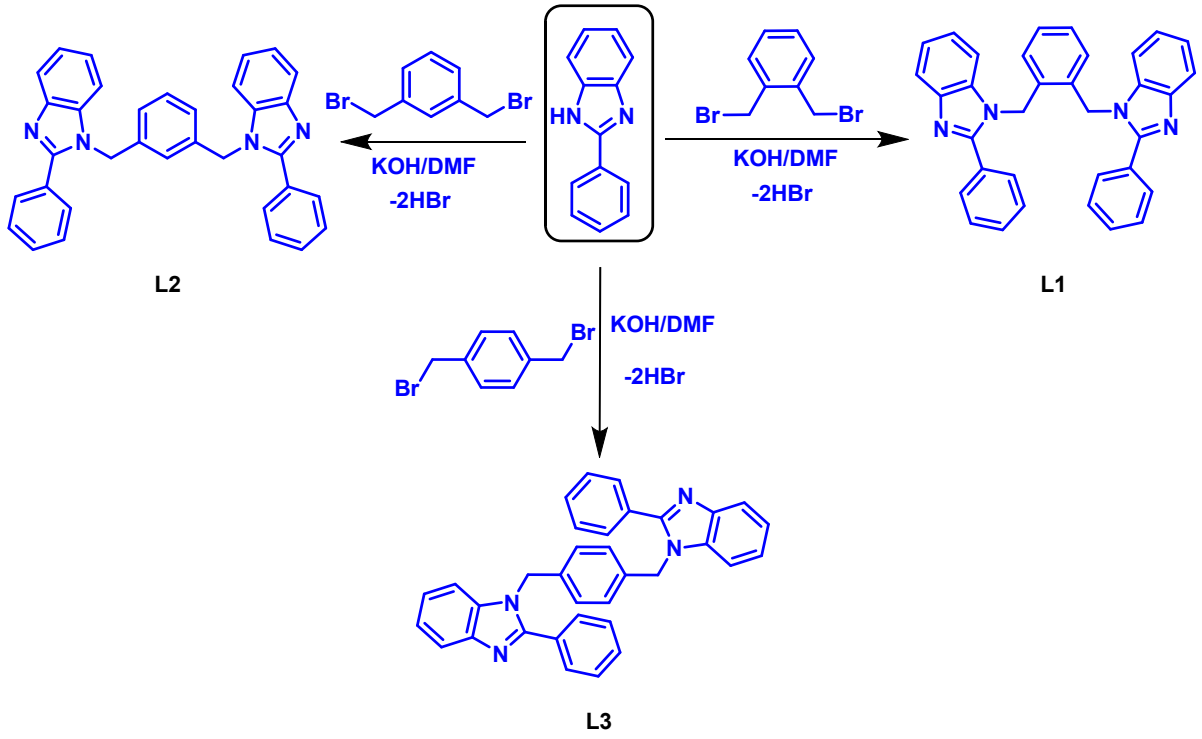
1. General Details

The solvents were dried by the usual methods. Chemicals 2-phenylbenzimidazole, 1,2-di(bromomethyl)benzene, 1,3-di(bromomethyl)benzene, 1,4-di(bromomethyl)benzene, sodium acetate anhydrous, ruthenium metal salt $[(\eta 6\text{-cymene})RuCl_2]_2$ were obtained from Sigma-Aldrich and used without further purification. Deuterated NMR solvents were purchased from Cambridge Isotope Laboratory (Andover, MA, USA). NMR spectra were recorded on Bruker 300 MHz spectrometer. ^{1}H NMR chemical shifts are reported relative to the residual protons of deuterated $CDCl_3$ (7.26 ppm) and deuterated DMSO-d_6 (2.50 ppm). ESI MS data of ligands and ruthenacycles were recorded on the Triple Quandrupole LC-Mass spectrometry (Finnigan TSQ Quantum Ultra EMR) using electrospray ionization and analyzed with the MassLynx software suite system at the Korea Basic Science Institute (Seoul).

2. Single crystal X-ray diffraction

Intensity data of suitably sized crystal of ruthenacycle **3** (***CCDC 1443147***) was collected at 100 K on an ADSC Quantum 210 CCD diffractometer with synchrotron radiation (λ = 0.70000 Å) at the Supramolecular Crystallography Beamline 2D, Pohang Accelerator Laboratory (PAL), Pohang, Korea. The raw data were processed and scaled using the program HKL3000. The structure was solved by direct methods, and the refinements were carried out with full-matrix least-squares on F^2 with appropriate software implemented in the SHELXL-2015 program.[S1] All the non-hydrogen atoms were refined anisotropically, and hydrogen atoms were added to their geometrically ideal positions. Three independent ruthenacycles **3** were observed in electron density map. Ruthenium atoms and the phenylbenximidazole rings in all three independent molecules are disordered over two positions. Crystal and structure refinement data summarized in Table S1.

Scheme S1: Synthesis of phenylbenzimidazolyle ditopic ligands **L1-L3**.



3.1. Synthesis of Ligand L1

A mixture of 2-phenylbenzimidazole (294 mg, 1.515 mmol) and KOH (170 mg, 3.031 mmol) in dimethlyformamide (DMF) (7 mL) were placed in a round-bottom flask and was allowed to stir at ~40 °C for 1 hr. Followed 1,2-di(bromomethyl)benzene (200 mg, 0.757 mmol) was added to the reaction mixture, which was allowed to stir continuously at ~40 °C for 24 h. The residue was poured into 100 mL of distilled water. The milky while precipitated was filtered and washed with excess of distilled water. Colorless crystalline materials were obtained from ethanol at room temperature after several days in 81% (299 mg) yield. ^{1}H NMR (300 MHz, $CDCl_3$): 5.34 (s, 4H, -CH_2-), 6.91–6.95 (m, 2H, Ar*H*), 7.07 (d, 2H, J = 9.0 Hz, Ar*H*), 7.22–7.25 (m, 1H, Ar*H*), 7.27–7.30 (m, 2H, Ar*H*), 7.33–7.36 (m, 2H, Ar*H*), 7.38–7.44 (m, 4H, Ar*H*), 7.46–7.51 (m, 2H,

Ar*H*), 7.58–7.61 (m, 4H, Ar*H*), 7.89 (d, 2H, *J* = 6.0 Hz, Ar*H*) and 8.08–8.11 (m, 1H, Ar*H*). Anal. Calcd for $C_{34}H_{26}N_4$ (Mwt. 490.59): C, 83.24; H, 5.34; N, 11.42. Found: C, 83.22; H, 5.34; N, 11.29. ESI-MS for **L1**: *m/z* = 491.10 $[M]^+$.

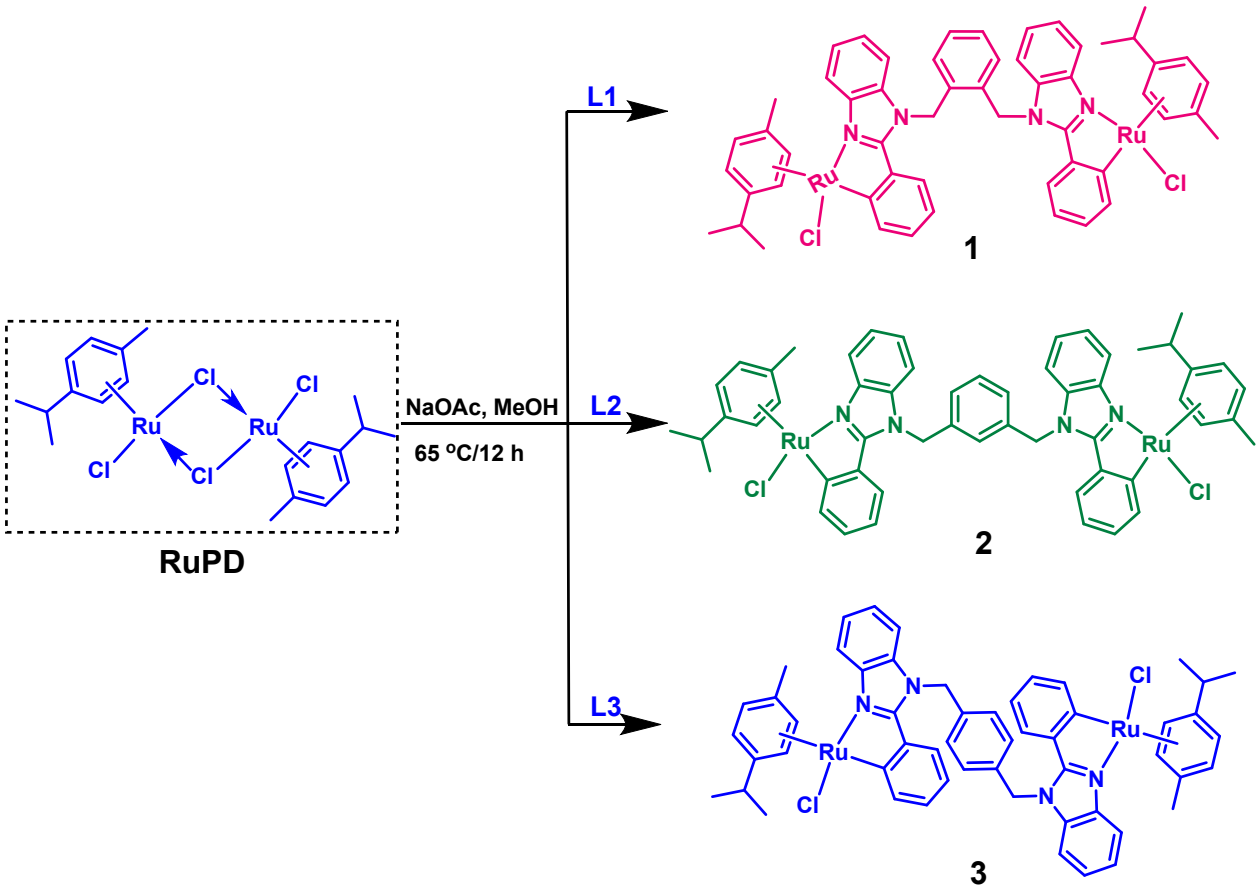
3.2. Synthesis of Ligand L2

The ligand **L2** was obtained by following the same procedure for **L1** using 2-phenylbenzimidazole (294 mg, 1.515 mmol), 1,3-di(bromomethyl)benzene (200 mg, 0.757 mmol) and KOH (170 mg, 3.031 mmol) in dimethlyformamide (DMF). White colored solid materials obtained from methanol after several days in 80 % (312 mg) yield. ^{1}H NMR (300 MHz, DMSO-d_6): 5.43 (s, 4H, -CH_2-), 6.52 (s, 1H, ArH), 6.93 (d, 2H, *J* = 6.0 Hz, Ar*H*), 7.17–7.39 (m, 11H, Ar*H*), 7.44–7.47 (m, 6H, Ar*H*) and 7.72 (d, 2H, *J* = 6.0 Hz, Ar*H*). Anal. Calcd for $C_{34}H_{26}N_4$ (Mwt. 490.59): C, 83.24; H, 5.34; N, 11.42. Found: C, 83.34; H, 5.36; N, 11.32. ESI-MS for **L2**: *m/z* = 491.19 $[M]^+$.

3.3. Synthesis of Ligand L3

The ligand **L3** was obtained by following the same procedure for **L1** using 2-phenylbenzimidazole (294 mg, 1.515 mmol), 1,4-di(bromomethyl)benzene (200 mg, 0.757 mmol) and KOH (170 mg, 3.031 mmol) in dimethlyformamide (DMF). White colored solid materials obtained from methanol after several days in 87 % (324 mg) yield. ^{1}H NMR (300 MHz, DMSO-d_6): 5.51 (s, 4H, -CH_2-), 6.91 (s, 4H, ArH), 7.18–7.26 (m, 4H, Ar*H*), 7.39–7.26 (m, 4H, Ar*H*), 7.48–7.51 (m, 6H, Ar*H*) and 7.65–7.71 (m, 6H, Ar*H*). Anal. Calcd for $C_{34}H_{26}N_4$ (Mwt. 490.59): C, 83.24; H, 5.34; N, 11.42. Found: C, 83.12; H, 5.41; N, 11.14. ESI-MS for **L3**: *m/z* = 491.21 $[M]^+$.

Scheme S2: Synthesis of Ruthenacycles **1**-**3**



4.1. Synthesis of Doubly Cyclometalated Ruthenacycle 1

One equivalent of ditopic phenylbenzimidazole derived ligand **L1** (50 mg, 0.081 mmol) was dissolved in freshly distilled methanol in dry round bottom flask equipped with stirrer and nitrogen atmosphere. A 2.3 equiv. of anhydrous sodium acetate (1.2 mmol) was added into at room temperature with constant stirring followed by addition of one equimolar [(η^6-cymene)$RuCl_2$]$_2$ (0.5 mmol). The reaction mixture was stirred at ~65 °C temperature for 12 h and the progress of reaction was monitored by TLC. After complete conversion, methanol was removed under reduced pressure and dried well under vacuum. Further, the solid was dissolved in dichloromethane (10 mL) and yellowish-brown solution was filtered and solution was

concentrated (~2 mL) and crystallized addition of methanol. The yellowish-brown crystalline product **1** was obtained after several days from chloroform and methanol in 91.9 % (77.32 mg) yield. Anal. Calcd for $C_{54}H_{52}Cl_2N_4Ru_2 \bullet CH_3OH$ (Mwt. 1062.11): C, 62.20; H, 5.31; N, 5.28. Found: C, 62.12; H, 5.25; N, 5.53. ^{1}H NMR (300 MHz, DMSO-d6) δ 0.76 (d, 6H, J = 6.0 Hz, C*H*$_3$-^{i}Pr), 0.82 (d, 6H, J = 6.0 Hz, C*H*$_3$-^{i}Pr), 2.06 (d, 6H, J = 6.0 Hz, C*H*$_3$), 2.11-2.19 (m, 2H, -C*H*-), 5.70 (d, 2H, J = 6.0 Hz, ArC*H*), 5.74-5.80 (m, 4H, ArC*H*), 6.06−6.09 (m, 2H, ArC*H*), 6.15-6.17 (m, 4H, ArC*H*/C*H*$_2$), 6.87-6.95 (m, 4H, ArC*H*), 7.02-7.10 (m, 3H, ArC*H*), 7.42-7.45 (m, 3H, ArC*H*), 7.49-7.53 (m, 4H, ArC*H*), 7.77 (d, 2H, J = 6.0 Hz, ArC*H*), 8.13 (d, 2H, J = 6 Hz, ArC*H*) and 8.29-8.34 (m, 2H, ArC*H*); ESI-MS for ruthenacycle **1**: *m/z* = 995.28 [**1**M - Cl]$^+$.

4.2. Synthesis of Doubly Cyclometalated Ruthenacycle 2

The ruthenacycle **2** was also synthesized by following procedure of **1**. One equivalent of ditopic phenylbenzimidazole derived ligand **L2** (50 mg, 0.081 mmol) was dissolved in freshly distilled methanol in dry round bottom flask equipped with stirrer and nitrogen atmosphere. A 2.3 equiv. of anhydrous sodium acetate (1.2 mmol) was added into at room temperature with constant stirring followed by addition of one equimolar [(η^6-cymene)$RuCl_2$]$_2$ (0.5 mmol). The reaction mixture was stirred at ~65 °C temperature for 12 h and the progress of reaction was monitored by TLC. After complete conversion, methanol was removed under reduced pressure and dried well under vacuum. Further, the solid was dissolved in dichloromethane (10 mL) and orange red-solution was filtered and solution was concentrated (~2 mL) and crystallized addition of methanol. The orange-red colored doubly cyclometalated ruthenacycle **2** was obtained in good yield 89.9 % (75.41 mg). Anal. Calcd for $C_{54}H_{52}Cl_2N_4Ru_2 \bullet CH_3OH + H_2O$ (Mwt. 1080.20): C, 61.16; H, 5.41; N, 5.19. Found: C, 60.86; H, 5.11; N, 5.18. ^{1}H NMR (300 MHz, DMSO-d6) δ 0.66 (d, 6H, J = 6.0 Hz, C*H*$_3$-iPr), 0.76 (d, 6H, J = 6.0 Hz, C*H*$_3$-iPr), 1.98 (s, 6H, C*H*$_3$), 2.06-2.15

(m, 2H, -C*H*-), 5.33 (d, 2H, J = 6.0 Hz, ArC*H*), 5.74 (d, 2H, ArC*H*), 5.66 (d, 2H, J = 6.0 Hz, ArC*H*), 5.86 (s, 4H, -CH_2-), 6.07 (d, 2H, J = 6.0 Hz, ArC*H*), 6.69-6.75 (m, 4H, ArC*H*), 6.97 (br, 1H), 7.04-7.09 (m, 3H, ArC*H*), 7.34 (t, 2H, J = 3 Hz, ArC*H*), 7.77 (t, 2H, J = 6.0 Hz, ArC*H*), 7.62 (d, 2H, J = 9 Hz, ArC*H*), 8.05 (d, 2H, J = 9.0 Hz, ArC*H*) and 8.30 (d, 2H, J = 9.0 Hz, ArC*H*); ESI-MS for ruthenacycle **2**: *m/z* = 995.13 [**2**M - Cl]$^+$.

4.3. Synthesis of Doubly Cyclometalated Ruthenacycle 3

The ruthenacycle **3** was also synthesized by following procedure of **1**. One equivalent of ditopic phenylbenzimidazole derived ligand **L3** (50 mg, 0.081 mmol) was dissolved in freshly distilled methanol in dry round bottom flask equipped with stirrer and nitrogen atmosphere. A 2.3 equiv. of anhydrous sodium acetate (1.2 mmol) was added into at room temperature with constant stirring followed by addition of one equimolar [(η^6-cymene)$RuCl_2$]$_2$ (0.5 mmol). The reaction mixture was stirred at ~65 °C temperature for 12 h and the progress of reaction was monitored by TLC. After complete conversion, methanol was removed under reduced pressure and dried well under vacuum. Further, the solid was dissolved in dichloromethane (10 mL) and yellowish-red solution was filtered and solution was concentrated (~2 mL) and crystallized addition of methanol. The yellowish-red crystalline product **3** was obtained after several days in 93.5 % (81.32 mg) yield. Anal. Calcd for $C_{54}H_{52}Cl_2N_4Ru_2 \bullet 2CH_3OH$ (Mwt. 1094.15): C, 61.47; H, 5.53; N, 5.12. Found: C, 61.75; H, 5.26; N, 5.51. ^{1}H NMR (300 MHz, DMSO-d6) δ 0.62 (d, 6H, J = 6.0 Hz, CH_3-iPr), 0.75 (d, 6H, J = 6.0 Hz, CH_3-iPr), 1.99 (s, 6H, CH_3), 2.05-2.11 (m, 2H, -C*H*-), 5.23 (d, 2H, J = 9.0 Hz, ArC*H*), 5.57 (d, 2H, J = 9.0 Hz, ArC*H*), 5.66 (t, 2H, J = 6.0 Hz, ArC*H*), 5.66 (d, 2H, J = 6 Hz, ArC*H*), 5.87 (s, 4H, -CH_2-), 6.03 (d, 2H, J = 6.0 Hz, ArC*H*), 6.86 (t, 2H, J = 9 Hz, ArC*H*), 6.93 (br, 2H, ArC*H*), 7.04 (t, 2H, J = 9.0 Hz, ArC*H*), 7.34 (t, 2H, J = 6 Hz, ArC*H*), 7.43 (t, 2H, J = 6.0 Hz, ArC*H*), 7.57 (d, 2H, J = 9 Hz, ArC*H*), 7.65 (d, 2H, J = 9.0 Hz,

ArC*H*), 7.85 (d, 2H, J = 9 Hz, ArC*H*) and 8.26 (d, 2H, J = 9.0 Hz, ArC*H*); ESI-MS for ruthenacycle **3**: *m/z* = 995.15 [**3**M - Cl]$^+$.

5. Biological Studies

5.1. Cancer cell growth inhibition test

AGS (human gastric carcinoma), SK-hep-1 (human hepatocellular carcinoma) and HCT-15 (human colorectal carcinoma) cell lines were routinely grown in RPMI 1640 and Dulbecco's Modified Eagle Medium (DMEM) supplemented with 10% heat inactivated fetal bovine serum (FBS) and 1% penicillin streptomycin at 37 °C and 5% CO_2. The cell suspensions were seeded into 96-well plates at a concentration of 1×10^4 cells/well. After incubation for 24 h, the cells were treated with each sample at concentrations of 0.8, 4, 20 and 100 μM for 24, 48 and 72 h. Stock solutions of 2 mg/mL compounds, oxaliplatin and doxorubicin were prepared in DMSO (dimethylsulfoxide) and stored at -20°C. Alternatively, cisplatin was dissolved in 0.9% NaCl (5 mM). MTT [3-(4,5-Dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide] solution was dissolved at 5 mg/mL in phosphate buffered saline (PBS, pH 7.2) and was filtered through a 0.22 μm Millipore filter. 10 μL of the MTT solution was added to each well. After incubation for 3 h at 37 °C and 5% CO_2, the MTT solution was then removed and 100 μL of DMSO was added to each well for cell lysis. The plates were read by a multi-reader (Tecan, Männedorf, Switzerland) at 550 nm for absorbance density values to determine the cell viability. The percentage of surviving cells was calculated from the ratio of the absorbance of treated to untreated cells. The half maximal inhibitory concentration (IC_{50}) values for the inhibition of cell growth were determined by fitting the plot of the logarithmic percentage of surviving cells against the logarithm of the drug concentration using a linear regression function.

5.2. RNA extract and first strand cDNA synthesis

Total RNA was extracted from AGS gastric carcinoma cells treated or non-treated with the samples using PureLink™ RNA Mini Kit ((Thermo Fisher Scientific, MA, USA) according to the manufacturer's instructions. cDNA synthesis was conducted by the reagents of GeneFishing DEG Premix Kit (Seegene, Seoul, Korea) except reverse transcriptase SuperScript II (Thermo Fisher Scientific, MA, USA). Three μg of total RNA was mixed with 2 μL of 10 μM cDNA synthesis primer dT-ACP1 (Seegene, Seoul, Korea) and DEPC-treated water to make the total volume of 9.5 μL. The mixture was incubated at 80°C for 3 min, and immediately placed on ice for 2 min before being briefly centrifuged. The mixture was added by 4 μL of 5× RT buffer (Noble Bio, Suwon, Korea), 5 μL of 2 mM dNTP (Takara, Osaka, Japan), 0.5 μL of 40 U/μL RNase inhibitor, and 1 μL of 200 U/μL M-MLV reverse transcriptase (Noble Bio, Suwon, Korea) with a final volume of 20 μL. cDNA synthesis was performed at 42°C for 90 min and the reaction was inactivated at 94°C for 2 min. After being incubated on ice for 2 min, the reaction was diluted five-fold by adding 80 μL of distilled water.

5.3. Annealing control primer (ACP)-based polymerase chain reaction (PCR)

Differentially expressed genes (DEGs) were screened using GeneFishing™ DEG kit (Seegene, Seoul, Korea) according to the manufacturer's instructions. Second-strand cDNA synthesis was conducted at 50°C for one cycle of first-stage PCR in a final reaction volume of 20 μL containing 3 μL of the diluted first-strand cDNA, 1 μL of 10 μM dT-ACP2, 10 μL of 2× SeeAmp™ ACP™ master mix, and 2 μL of 5 μM arbitrary ACP. PCR for second-strand synthesis was performed in one cycle of incubation at 94°C for 5 min, 50°C for 3 min, and 72°C

for 1 min. After the completion of second-strand cDNA synthesis, 40 cycles of amplification were performed with denaturation at 94°C for 40 sec, annealing at 65°C for 40 sec, and extension at 72°C for 40 sec; this was followed by a final extension step of 5 min at 72°C.

PCR products were separated in 2% agarose gel with 0.5× TAE buffer, and differentially expressed cDNA bands were excised from the gels and purified**. DNA sequencing** was performed by **SolGent** Co. (Daejeon, Korea), and the sequences were analyzed using BLASTX search program at the National Center for Biotechnology Information (NCBI) GenBank.

5.4. Quantitative RT-PCR analysis

Total RNA was extracted from AGS gastric carcinoma cells using PureLink™ RNA Mini Kit. One μg of total RNA was reverse transcribed in a 20 μL volume using oligo (dT) primers, with enzyme and buffer supplied in the PrimeScript II 1st strand cDNA Synthesis kit (Takara, Osaka, Japan). Quantitative real-time PCR reactions were performed on a MX3005P (Stratagene, CA, USA) using the following primers; RPS21 was 5'-GCTGCTTCCTTTCTCTCTCTG-3', 5'-GCCTGTGACCTTGTCAACCT-3' and β-actin was 5'-GTCCACCGCAAATGCTTCTA-3', 5'-TGCTGTCACCTTCACCGTTC-3'. For real-time PCR, SYBR Premix Ex Taq II (Takara, Osaka, Japan) was used. The final volume of the reaction was 25 μL containing 2 μL cDNA template, 12.5 μL Master Mix, 1 μL each primer (10 μM stock solution), and 8.5 μL sterile distilled water. The thermal cycling profile consisted of a pre-incubation step at 95°C for 10 min, followed by 40 cycles of 95°C (30 s), 53°C (60 s) and 72°C (30 s). Relative quantitative evaluation of RPS21 gene levels was performed by the comparative CT (cycle threshold) method.

5.5. ELISA (Enzyme-linked immunosorbent assay) array

For determination of secretion of 31 cytokines (TNF-α, IFNγ, G-CSF, GM-CSF, IL-1α, IL-8, IP-10, Rantes, VEGF, EGF, IL-6, Resistin, PAI-1, IL-12, IL-13, Eotaxin-3, PDGF-BB, PIGF-1, β-NGF, SCF, MCP-1, MIP-1α, IL-2, IL-4, IL-10, FGFβ, Leptin, IGF-1, TGF-β, Adipo and IL-17α), THP-1 human monocytes were incubated with doubly cyclometalated ruthenacycle **2** (10 μM), or only medium for 24 h. The content of 31 cytokines was determined using a Human Cytokine ELISA Plate Array I kit (Signosis, CA, USA). The assay was carried out according to the manufacture instructions. Chemiluminescent detection was performed with the Multi-reader.

6. References

S1. G. Sheldrick, *Acta Crustallogr. Sect. A* 2008, *A64*, 112.

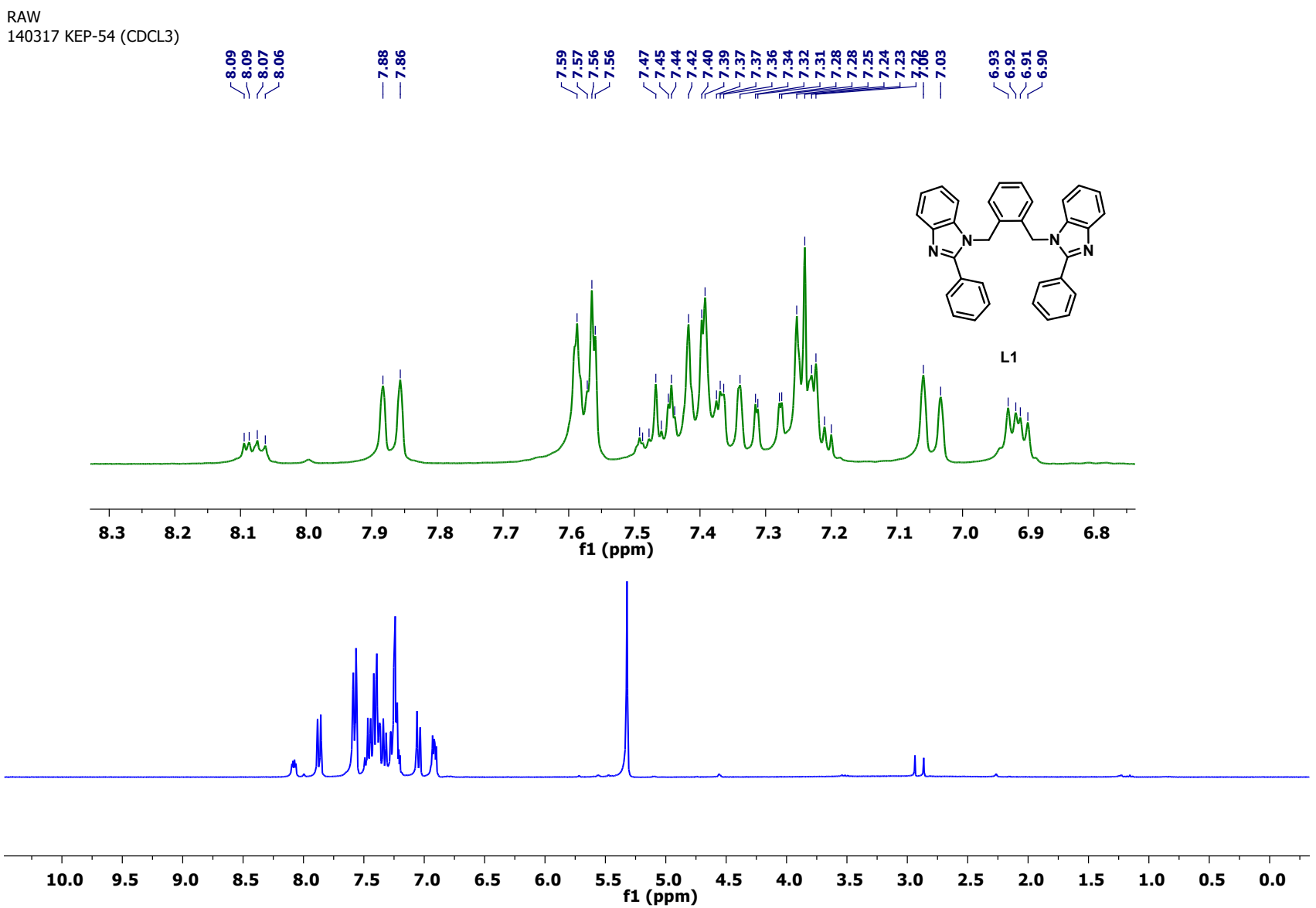


Figure S1: ^{1}H NMR spectrum of ligand **L1** in $CDCl_3$ in 300 MHz.

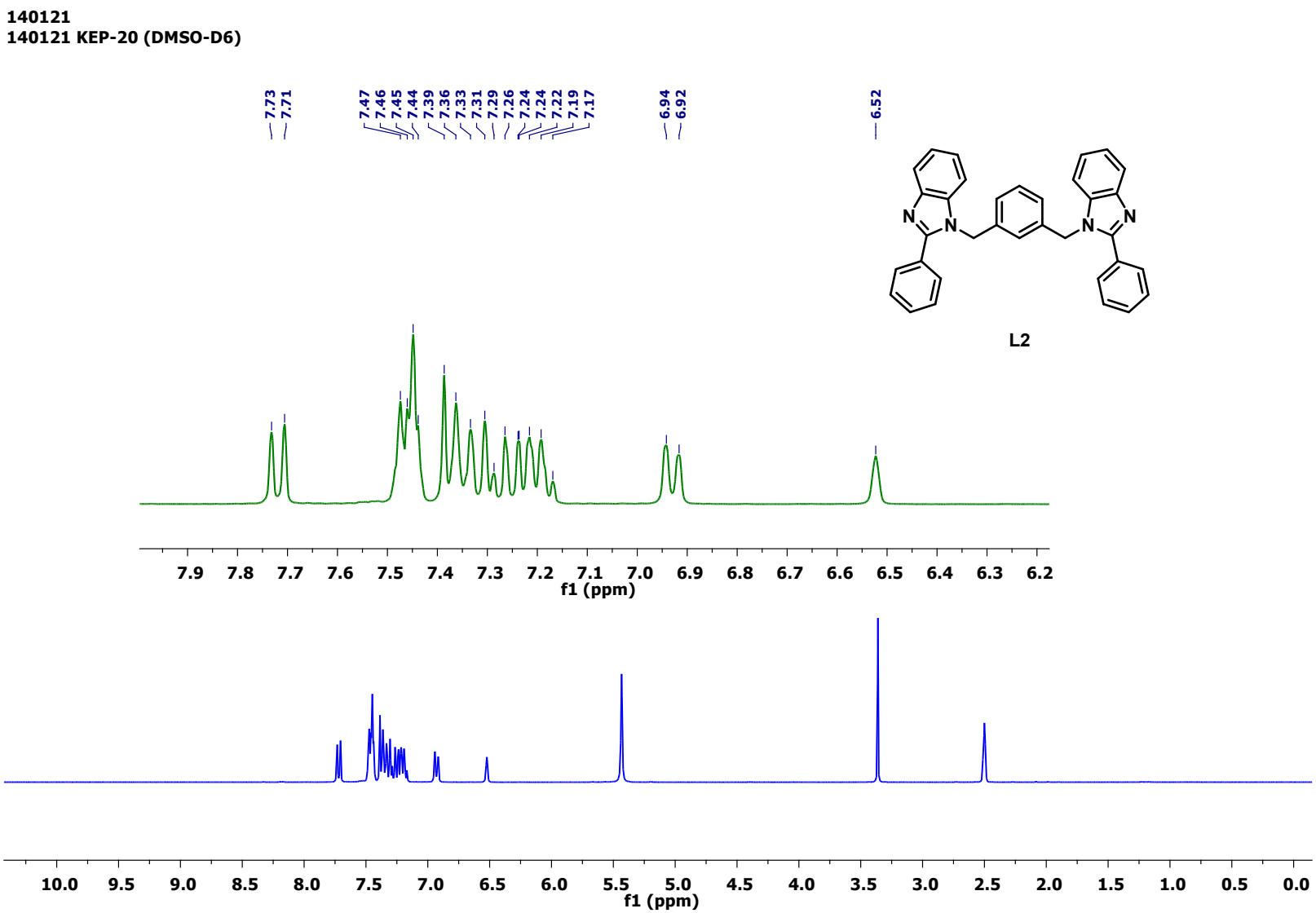


Figure S2: ^{1}H NMR spectrum of ligand **L2** in DMSO-d_6 in 300 MHz.

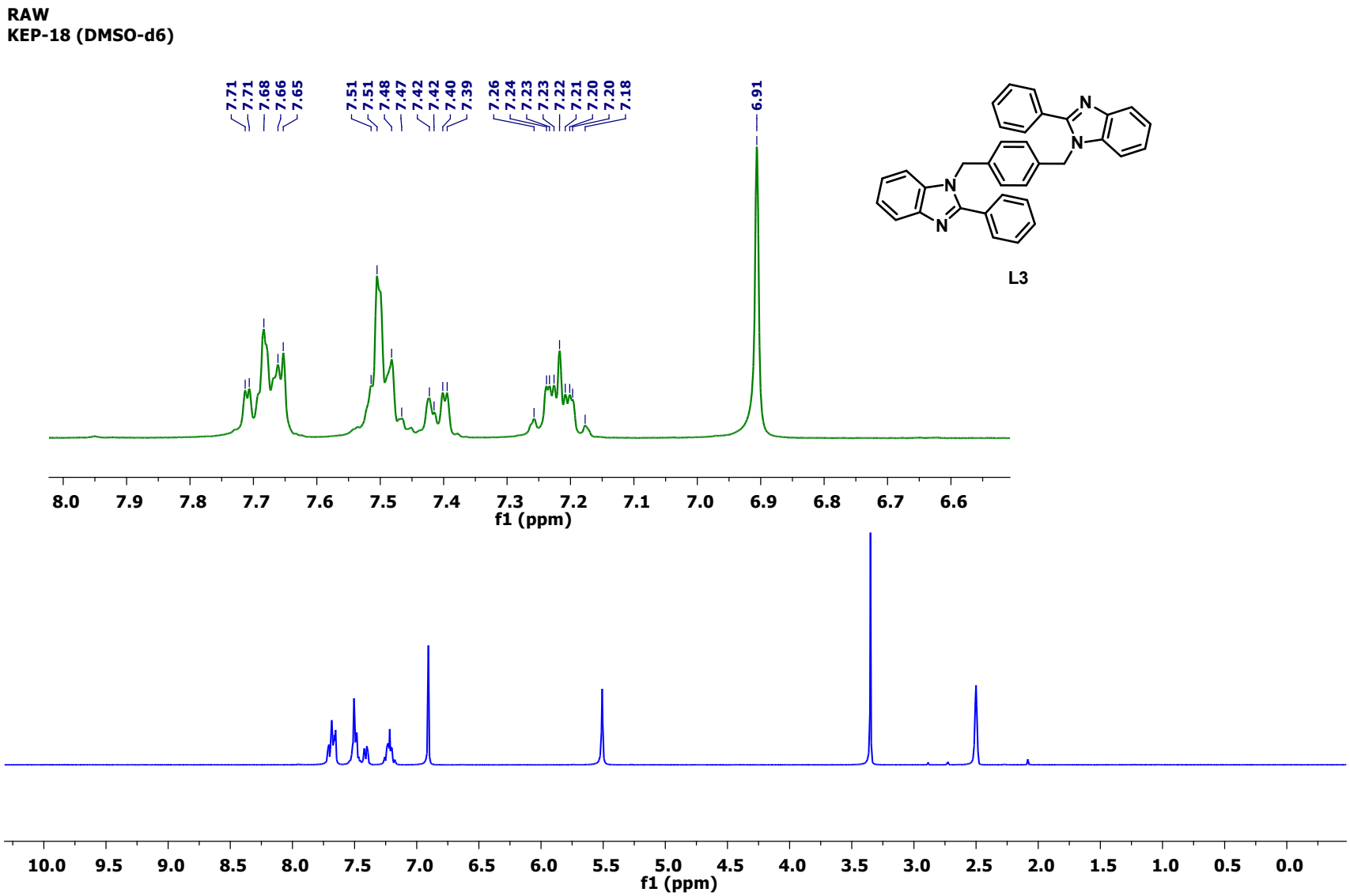


Figure S3: ^{1}H NMR spectrum of ligand **L3** in DMSO-d_6 in 300 MHz.

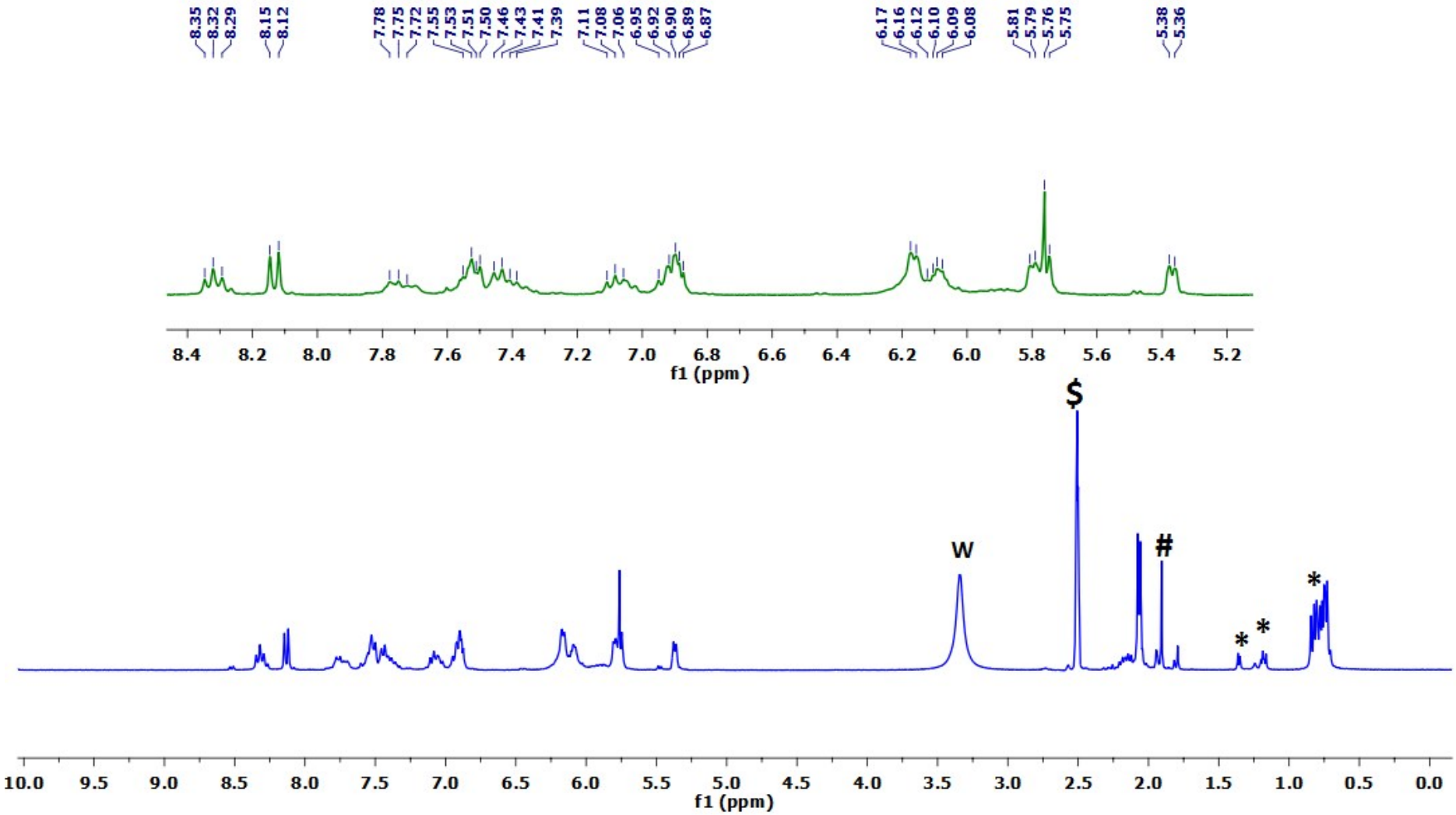


Figure S4: [1]H NMR spectrum of doubly cyclometalated ruthenacycle **1** in DMSO-d_6 in 300 MHz, the NMR solvent impurities labeled as $, DMSO; w, water; *, grease/hexane and #, acetone.

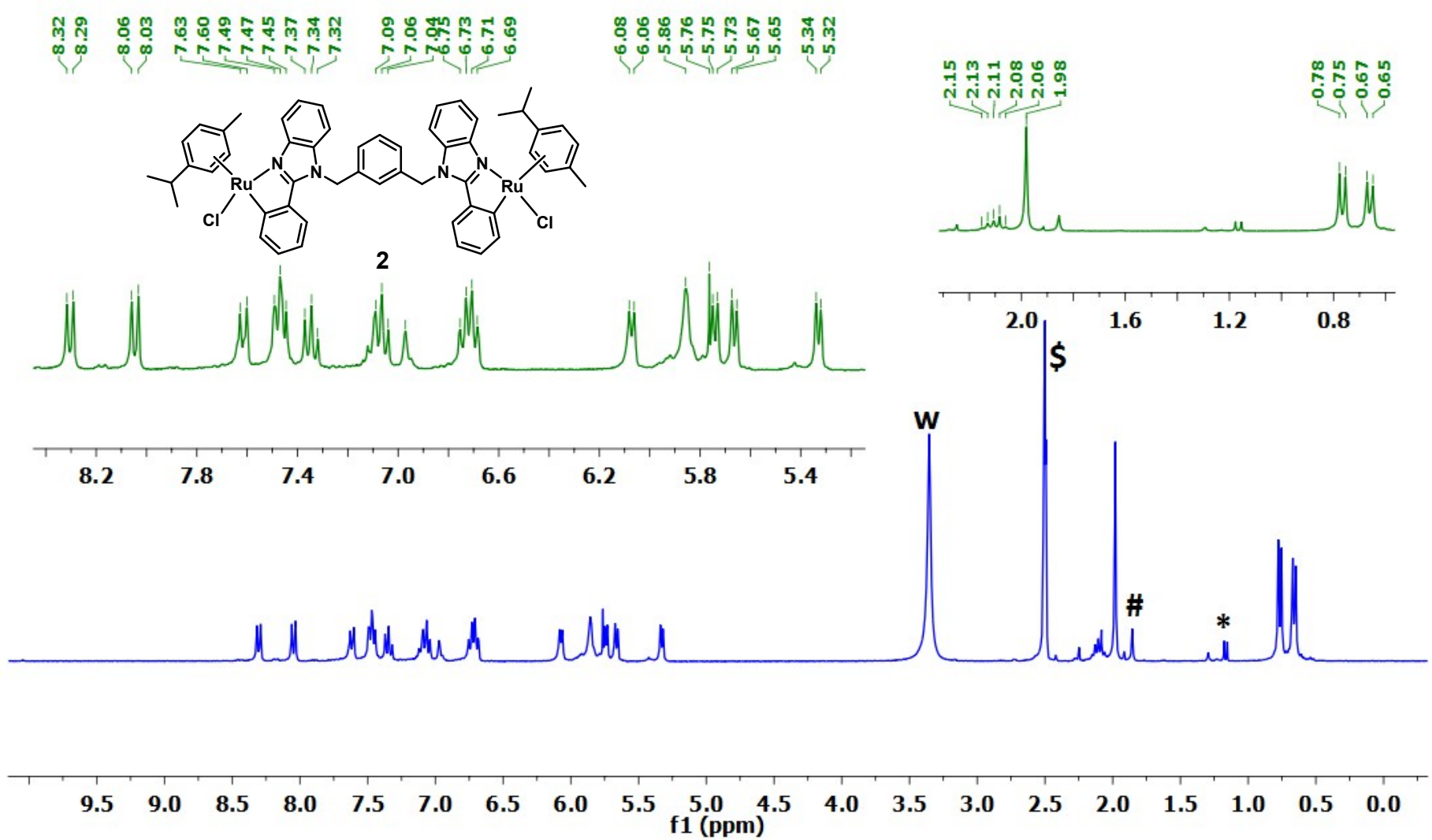


Figure S5: [1]H NMR spectrum of doubly cyclometalated ruthenacycle **2** in DMSO-d_6 in 300 MHz, the NMR solvent impurities labeled as $, DMSO; w, water; *, grease and #, acetone.

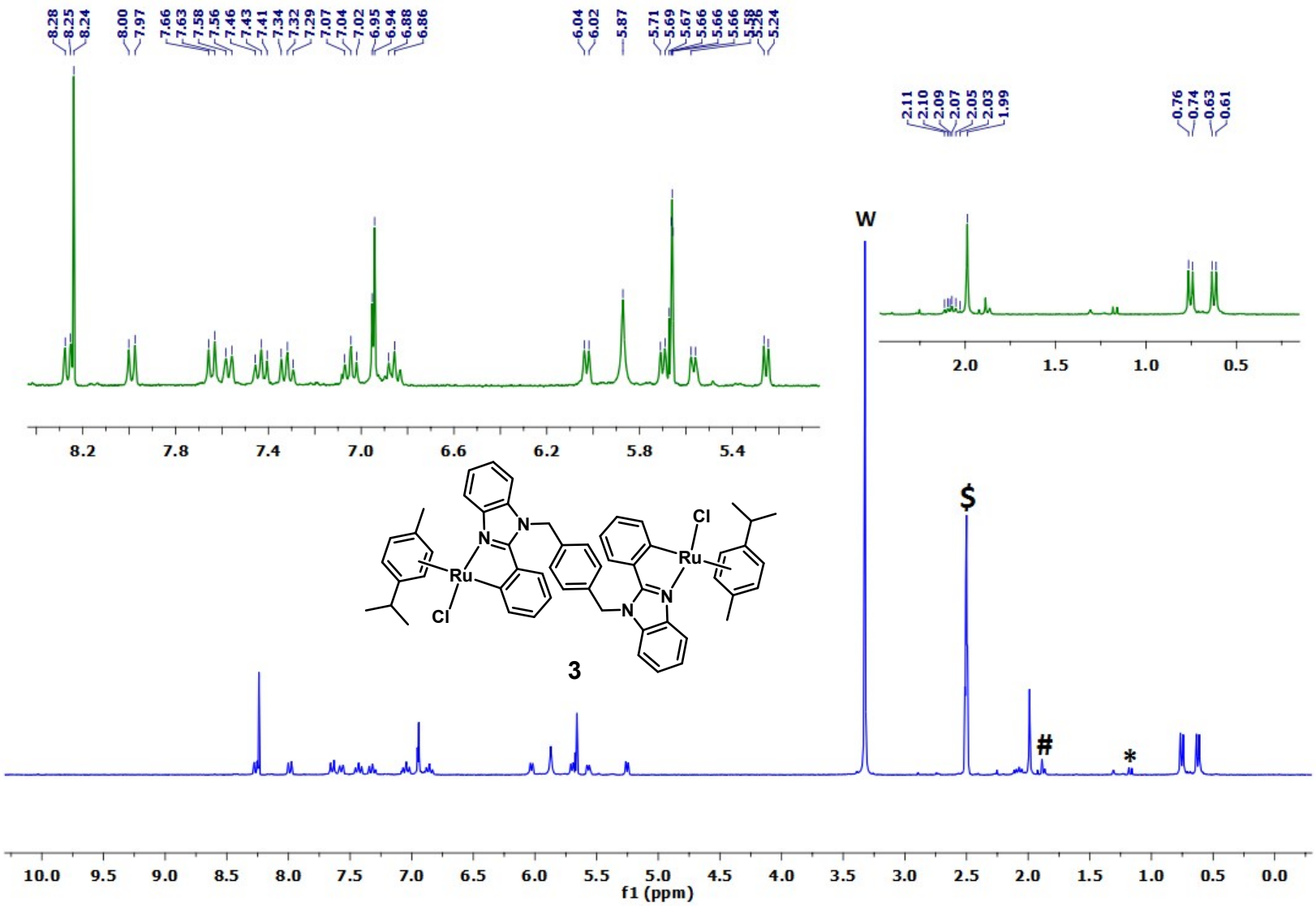


Figure S6: [1]H NMR spectrum of doubly cyclometalated ruthenacycle **3** in DMSO-d_6 in 300 MHz, the NMR solvent impurities labeled as $, DMSO; w, water; *, grease and #, acetone.

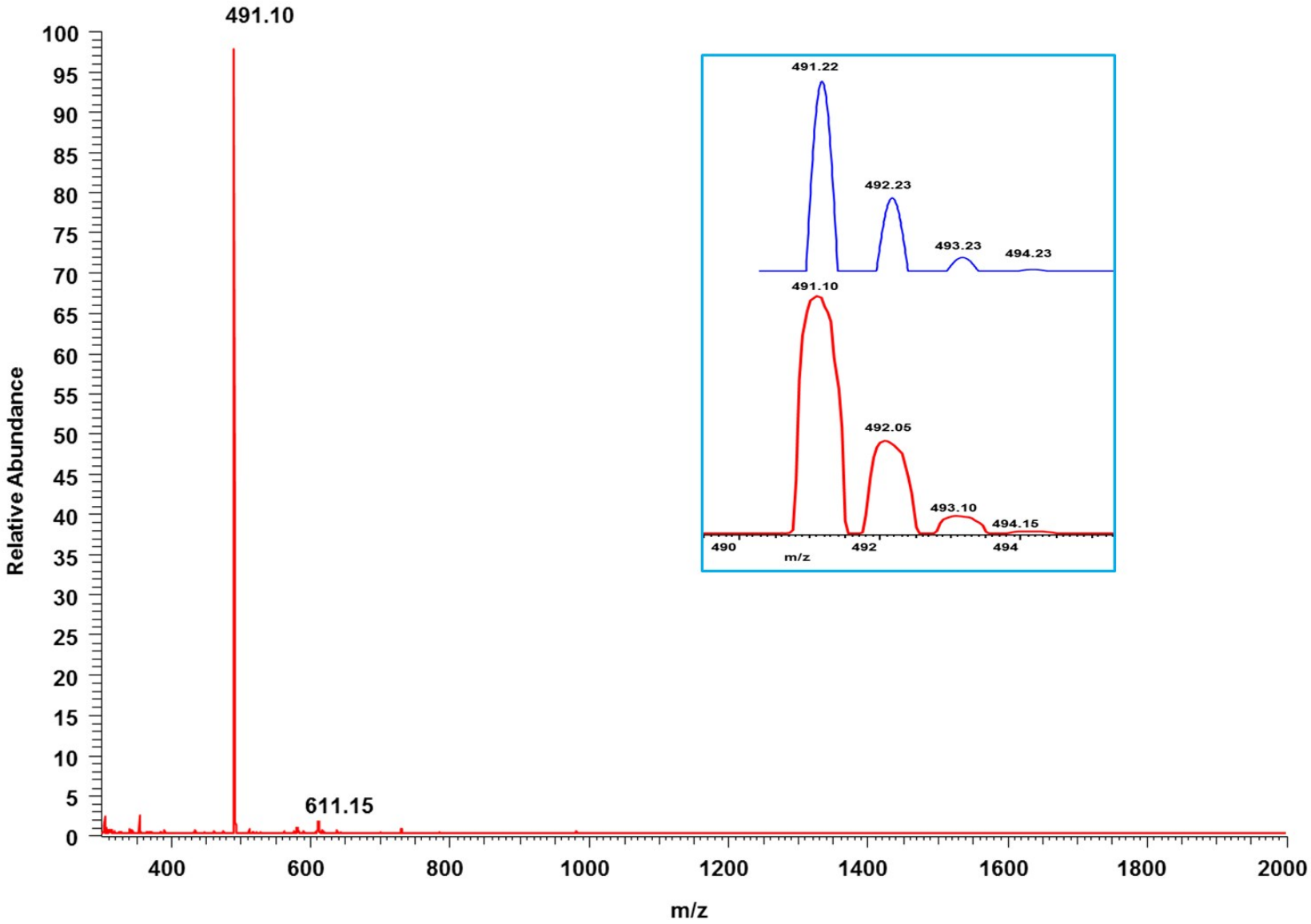


Figure S7. ESI-MS spectrum of ligand **L1.** *Insert:* Calculated (blue) and experimental (red) [**L1**]$^{+}$

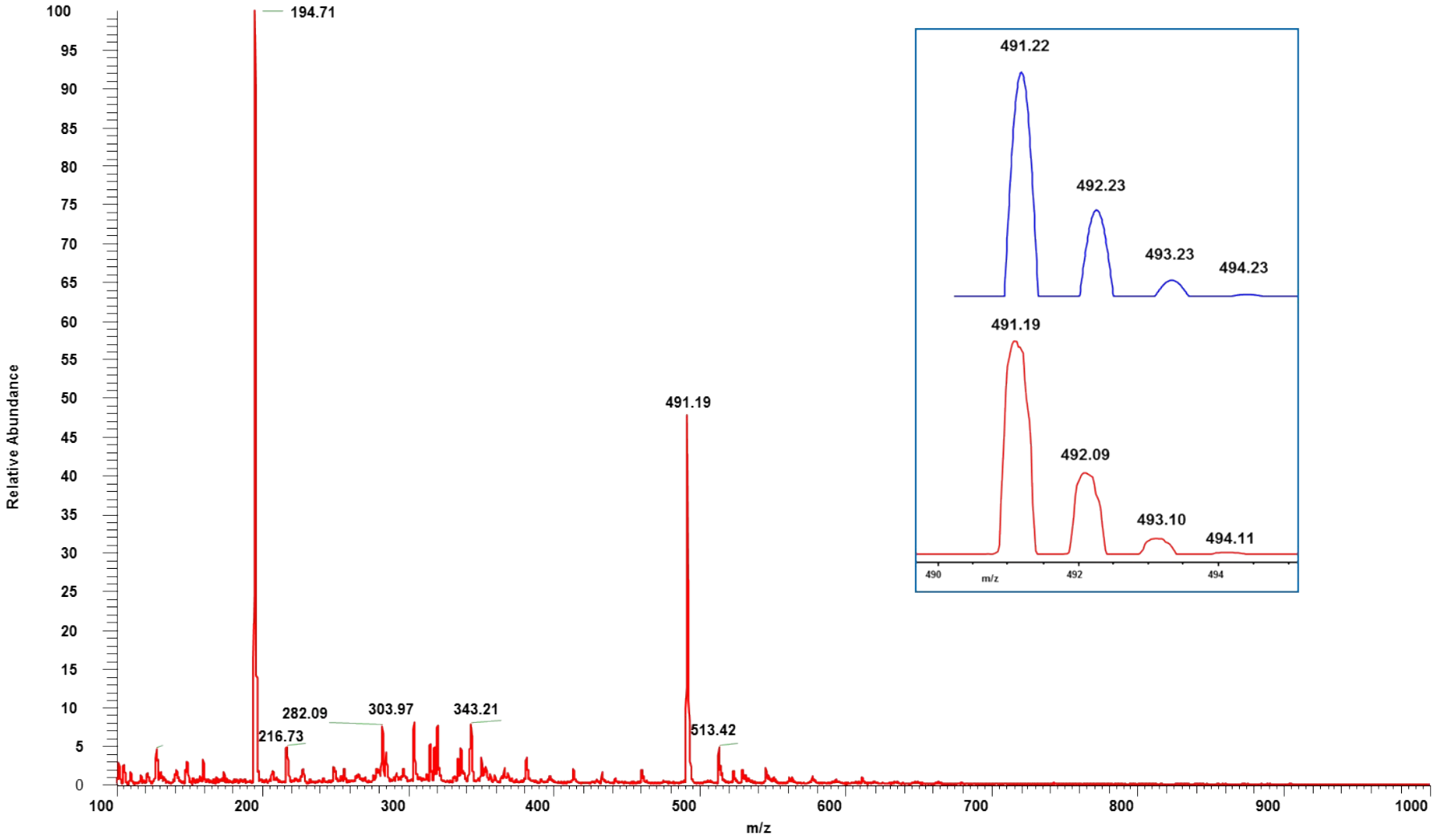


Figure S8. ESI-MS spectrum of ligand **L2.** *Insert:* Calculated (blue) and experimental (red) [**L2**]$^{+}$

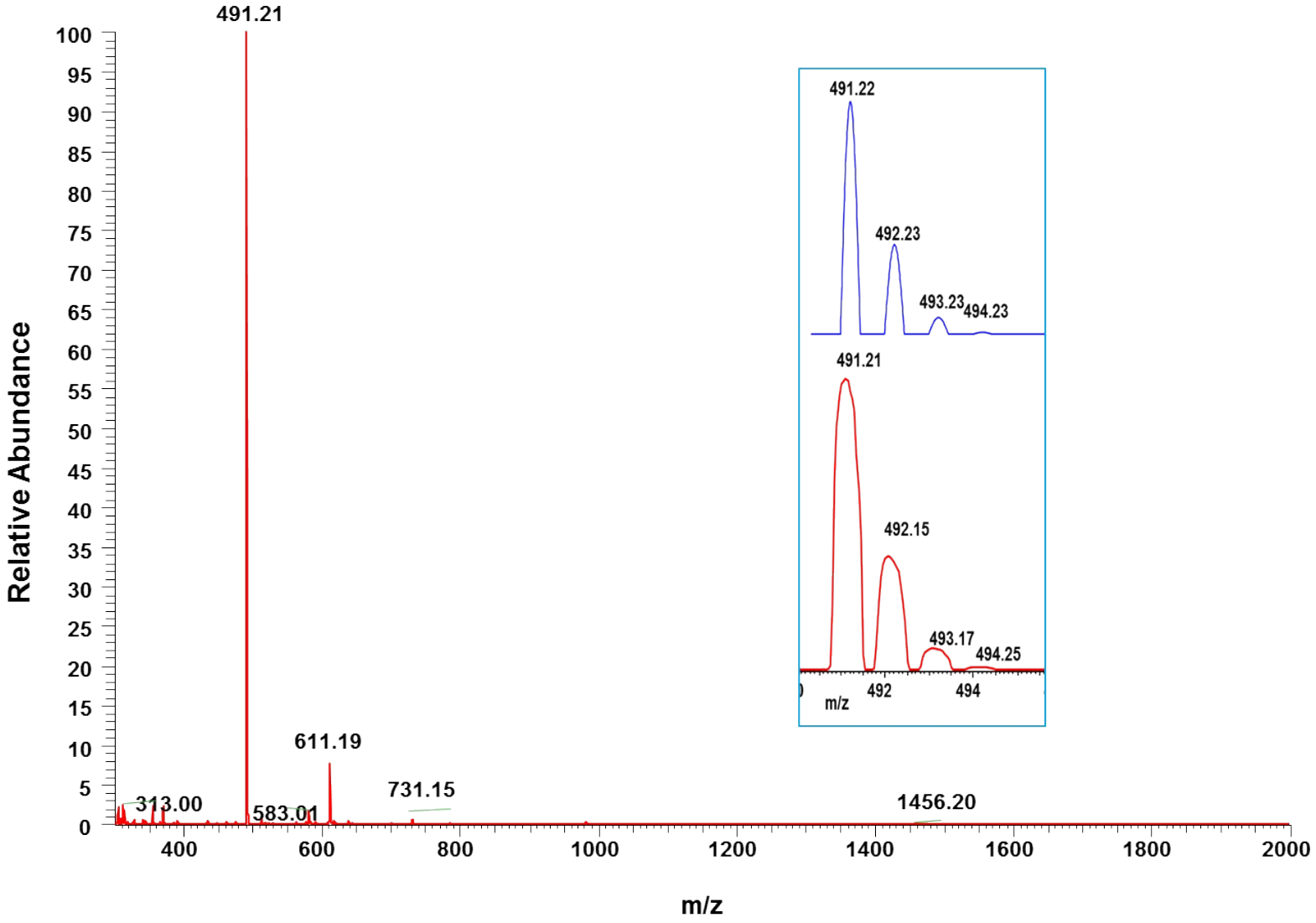


Figure S9. ESI-MS spectrum of ligand **L3.** *Insert:* Calculated (blue) and experimental (red) [**L3**]$^{+}$

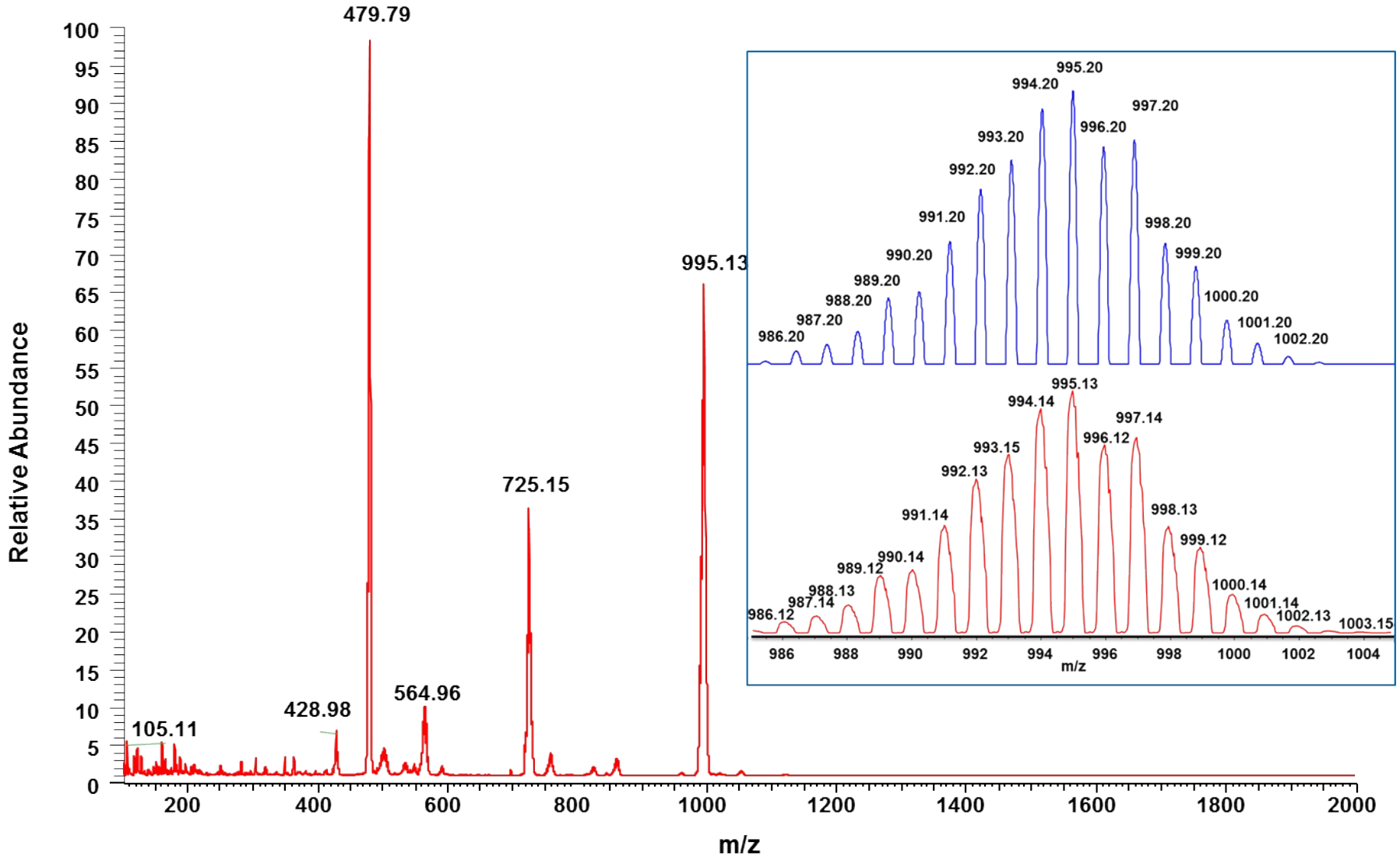


Figure S10: ESI-MS spectrum of ruthenacycle **2.** *Insert:* Calculated (blue) and experimental (red) ESI-MS spectra of ruthenacycle [**2**M–Cl]$^+$

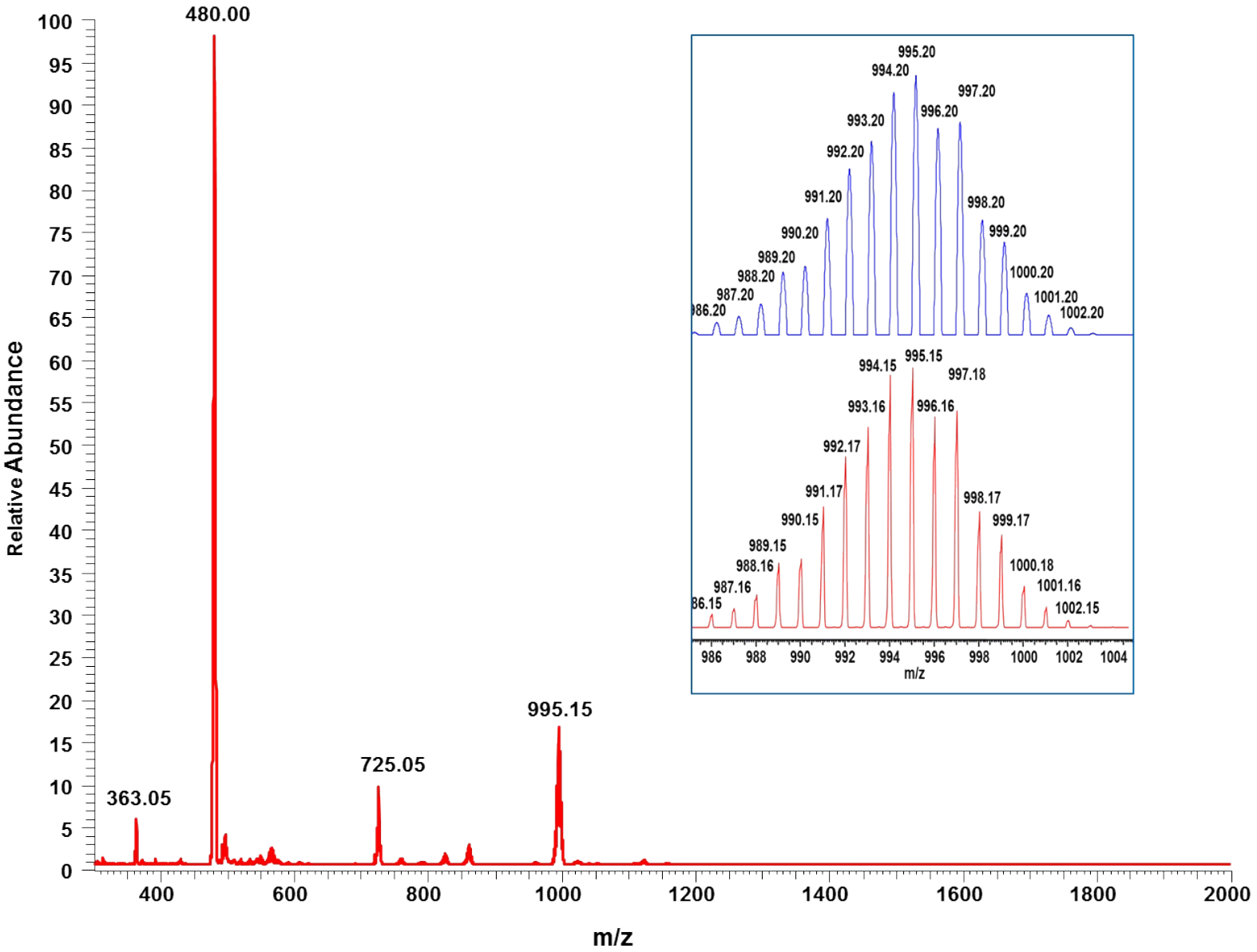


Figure S11: ESI-MS spectrum of ruthenacycle **3.** *Insert:* Calculated (blue) and experimental (red) ESI-MS spectra of ruthenacycle [**3**M–Cl]$^+$

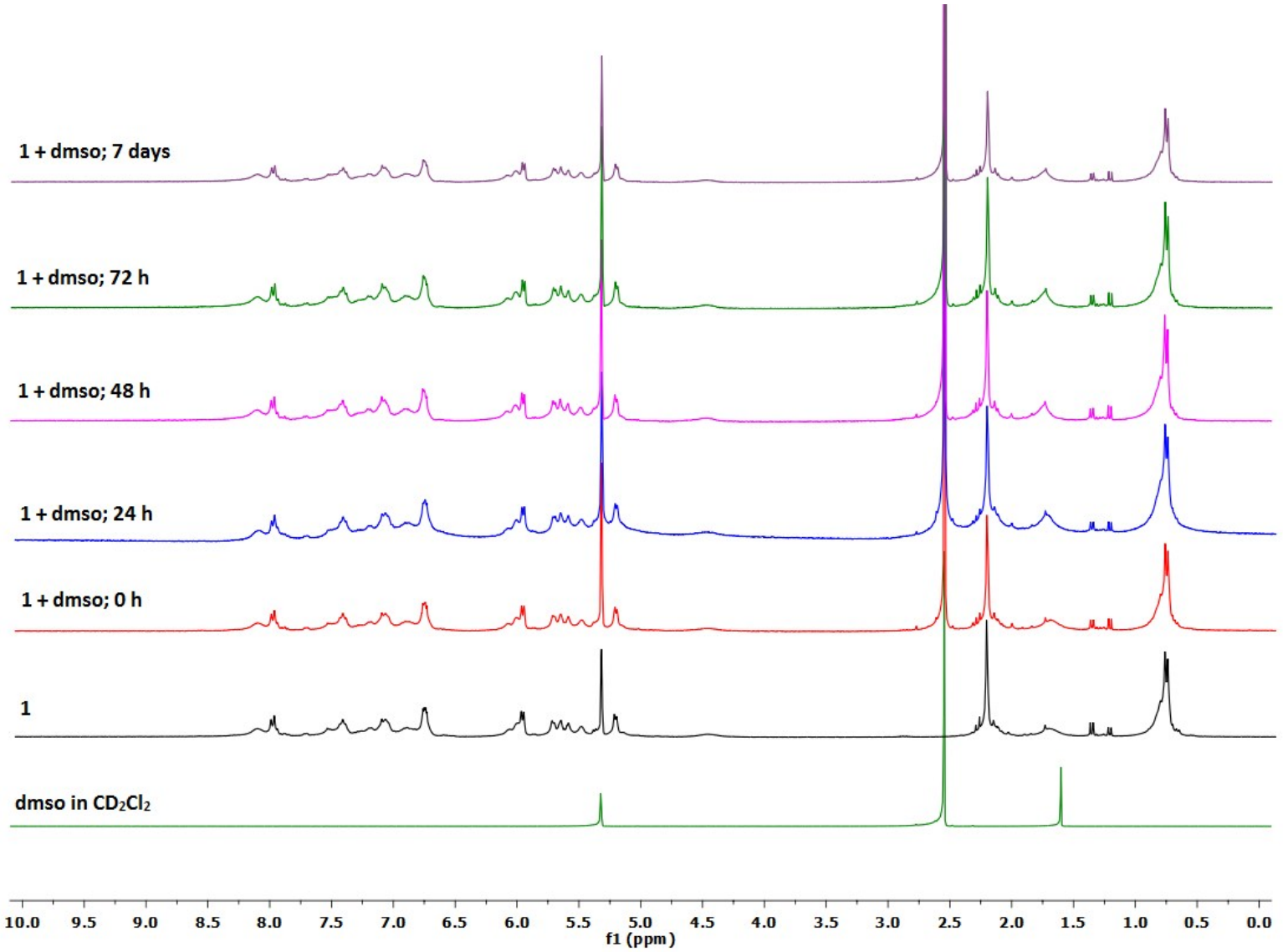


Figure S12: ^{1}H NMR spectra of DMSO reactivity study with ruthenacycle **1** in CD_2Cl_2 at room temperature.

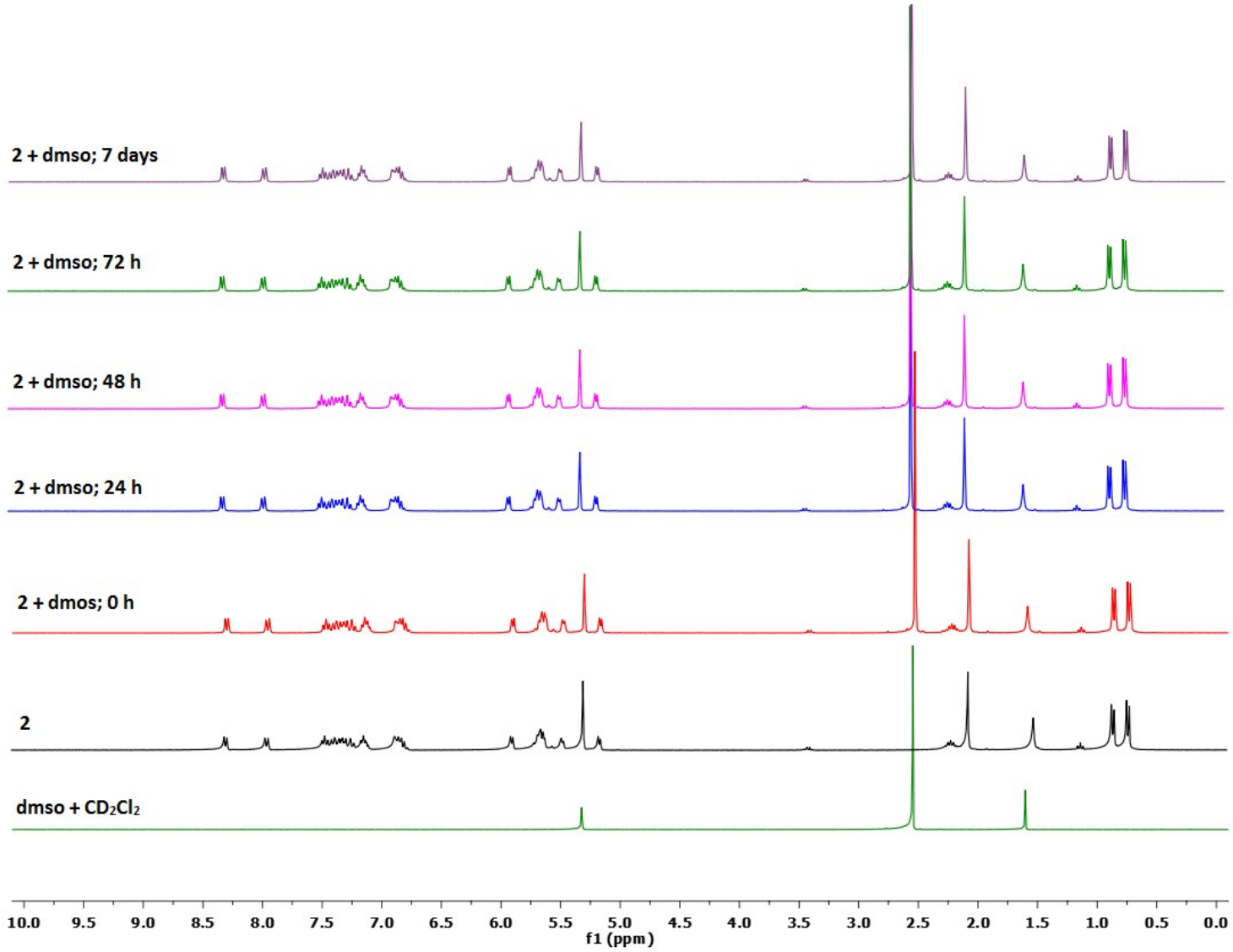


Figure S13: ^{1}H NMR spectra of DMSO reactivity study with ruthenacycle **2** in CD_2Cl_2 at room temperature.

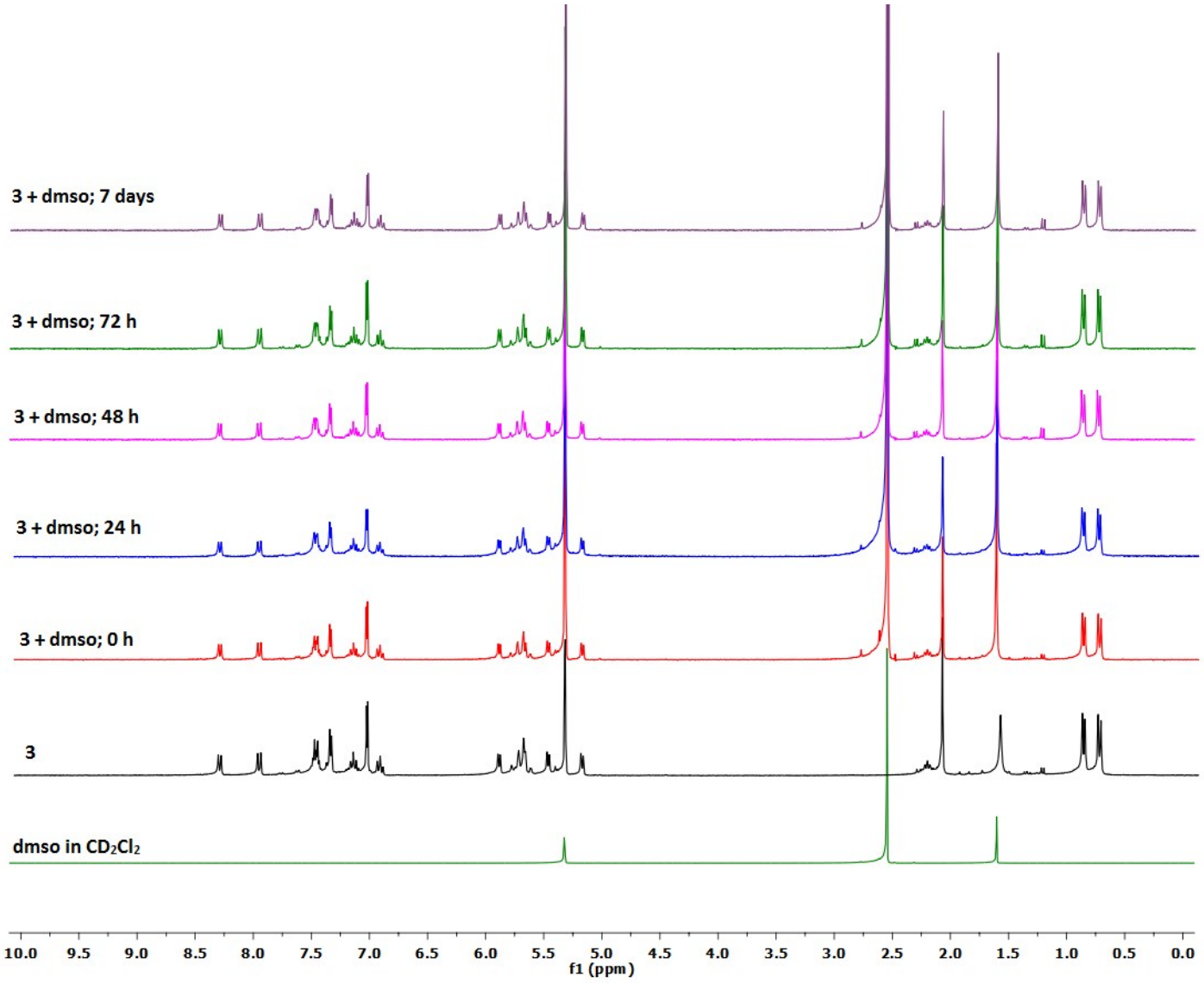


Figure S14: ^{1}H NMR spectra of DMSO reactivity study with ruthenacycle **3** in CD_2Cl_2 at room temperature.

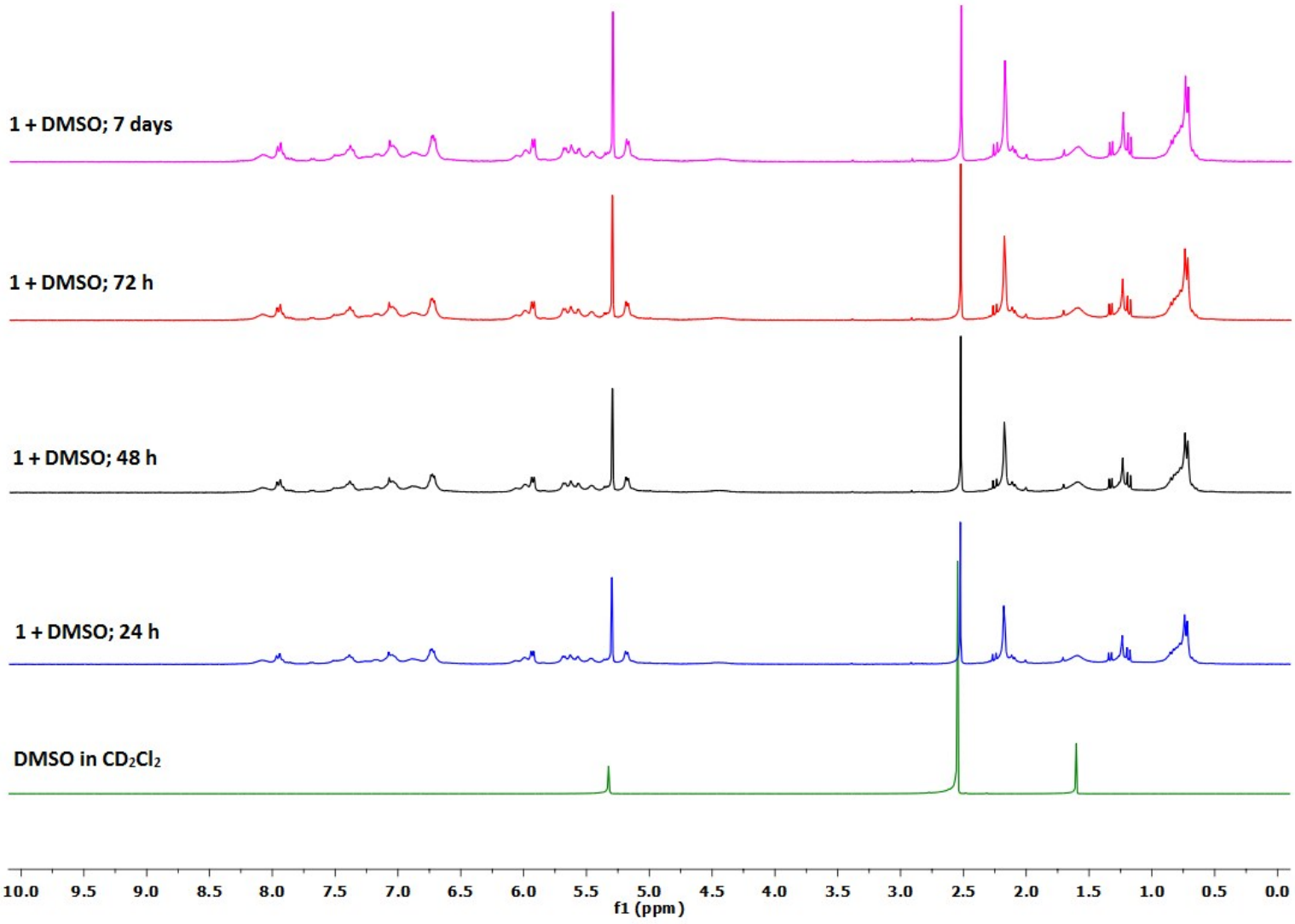


Figure S15: ^{1}H NMR spectra of DMSO reactivity study with ruthenacycle **1** in CD_2Cl_2 at at 50 ^{o}C.

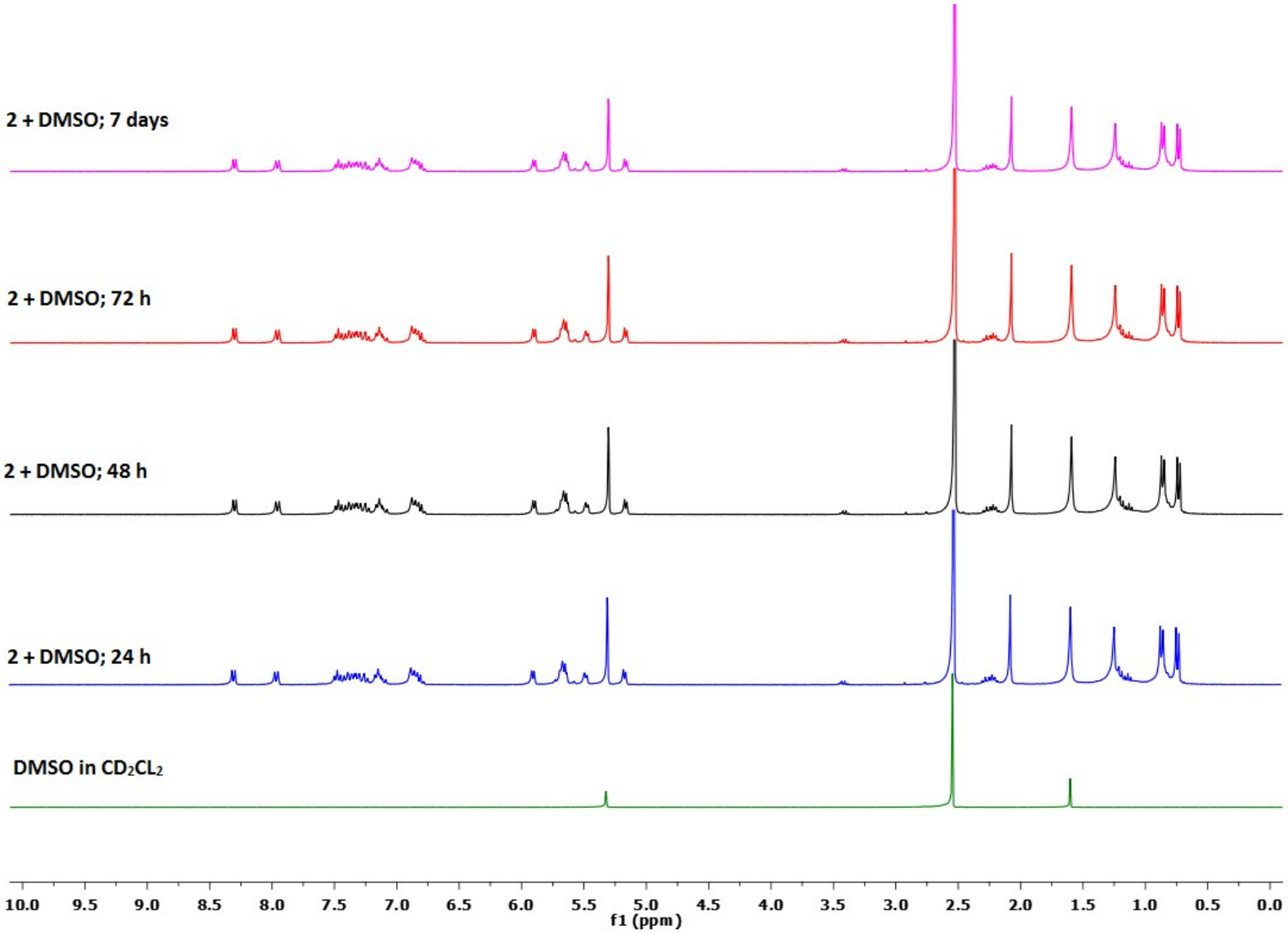


Figure S16: ^{1}H NMR spectra of DMSO reactivity study with ruthenacycle **2** in CD_2Cl_2 at at 50 ^{o}C.

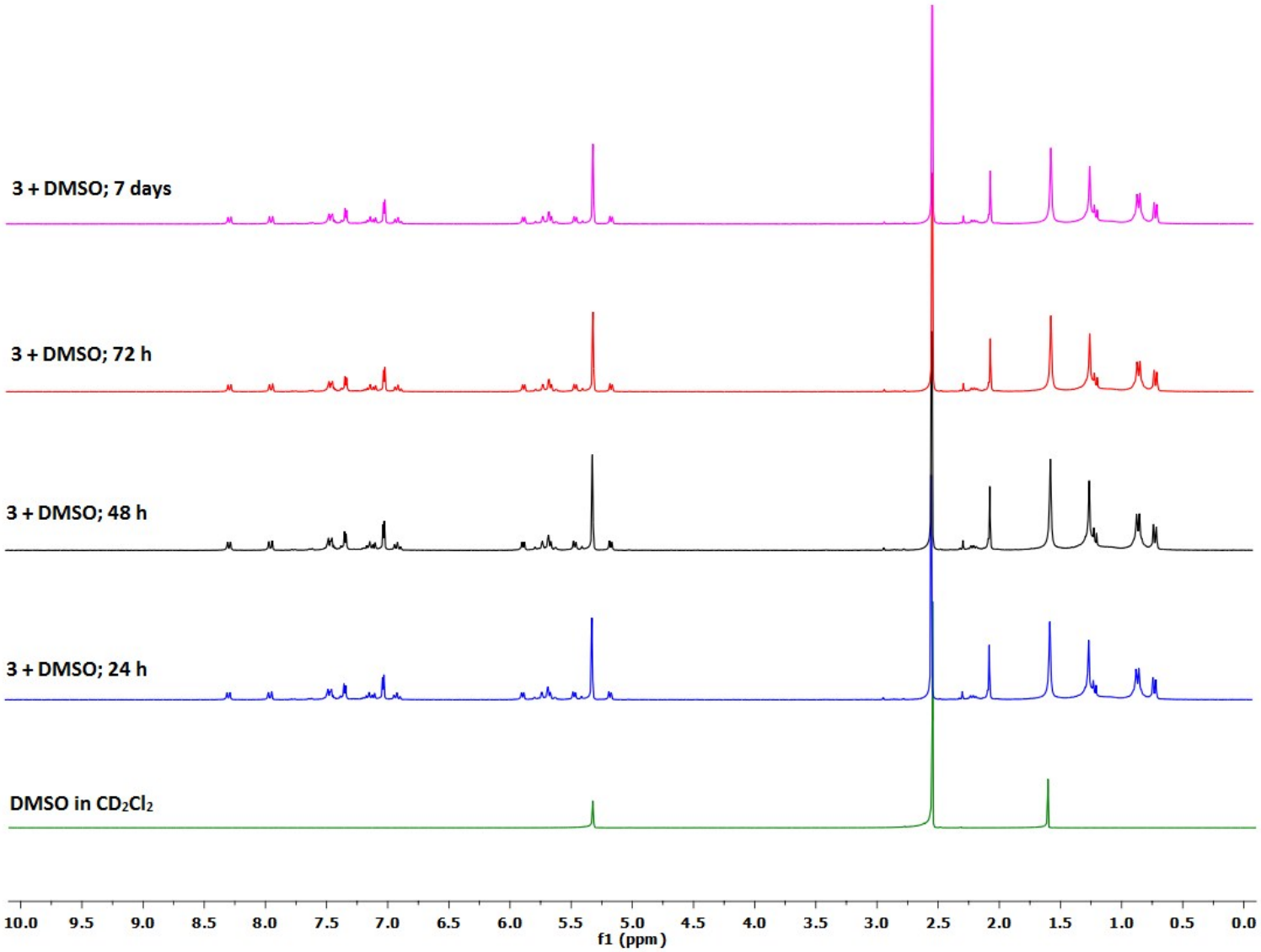


Figure S17: ^{1}H NMR spectra of DMSO reactivity study with ruthenacycle **3** in CD_2Cl_2 at at 50 ^{o}C.

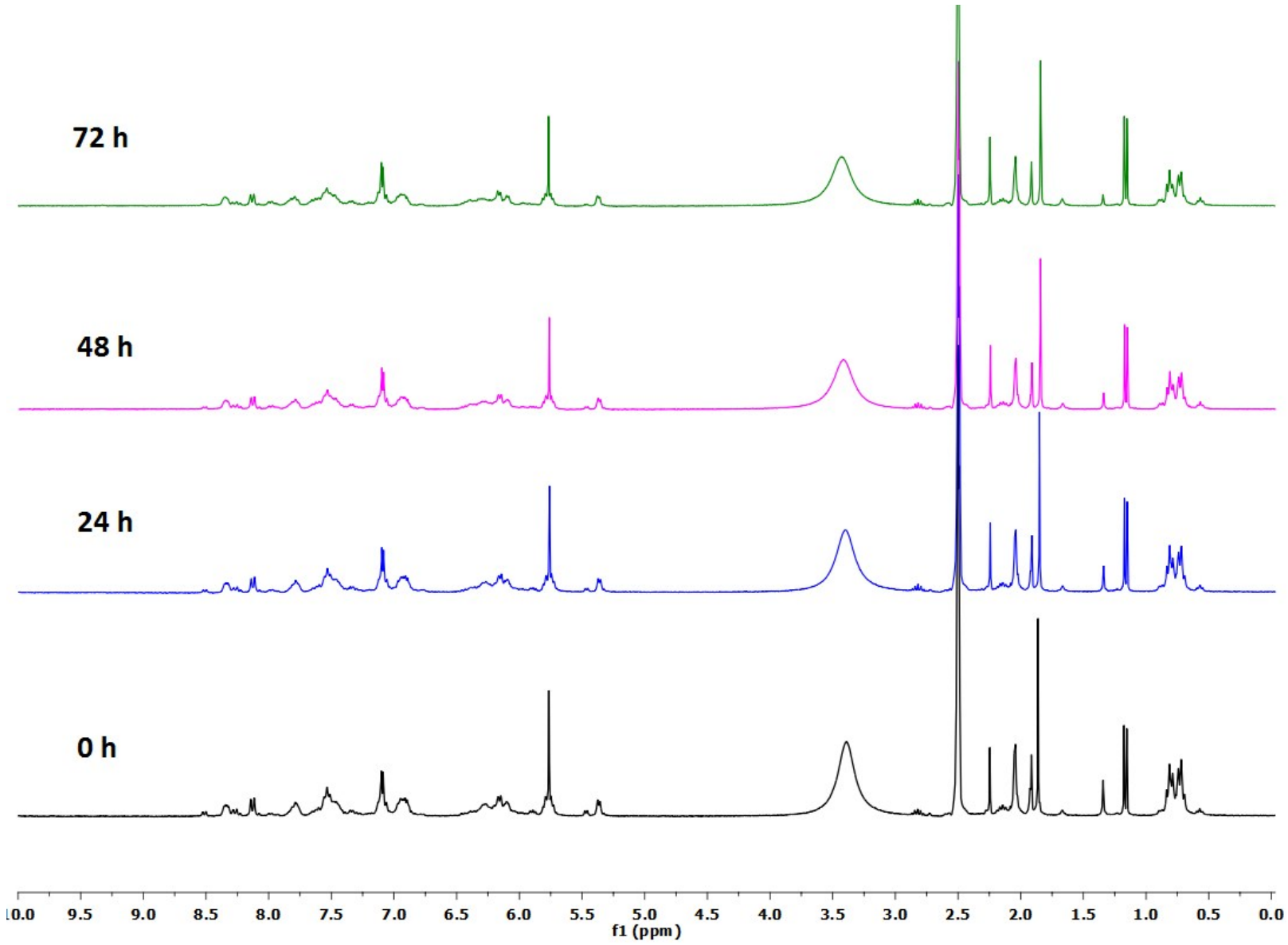


Figure S18: Time-dependent ^{1}H NMR spectra of ruthenacycle **1** in DMSO-d_6 from 0 to 72 h.

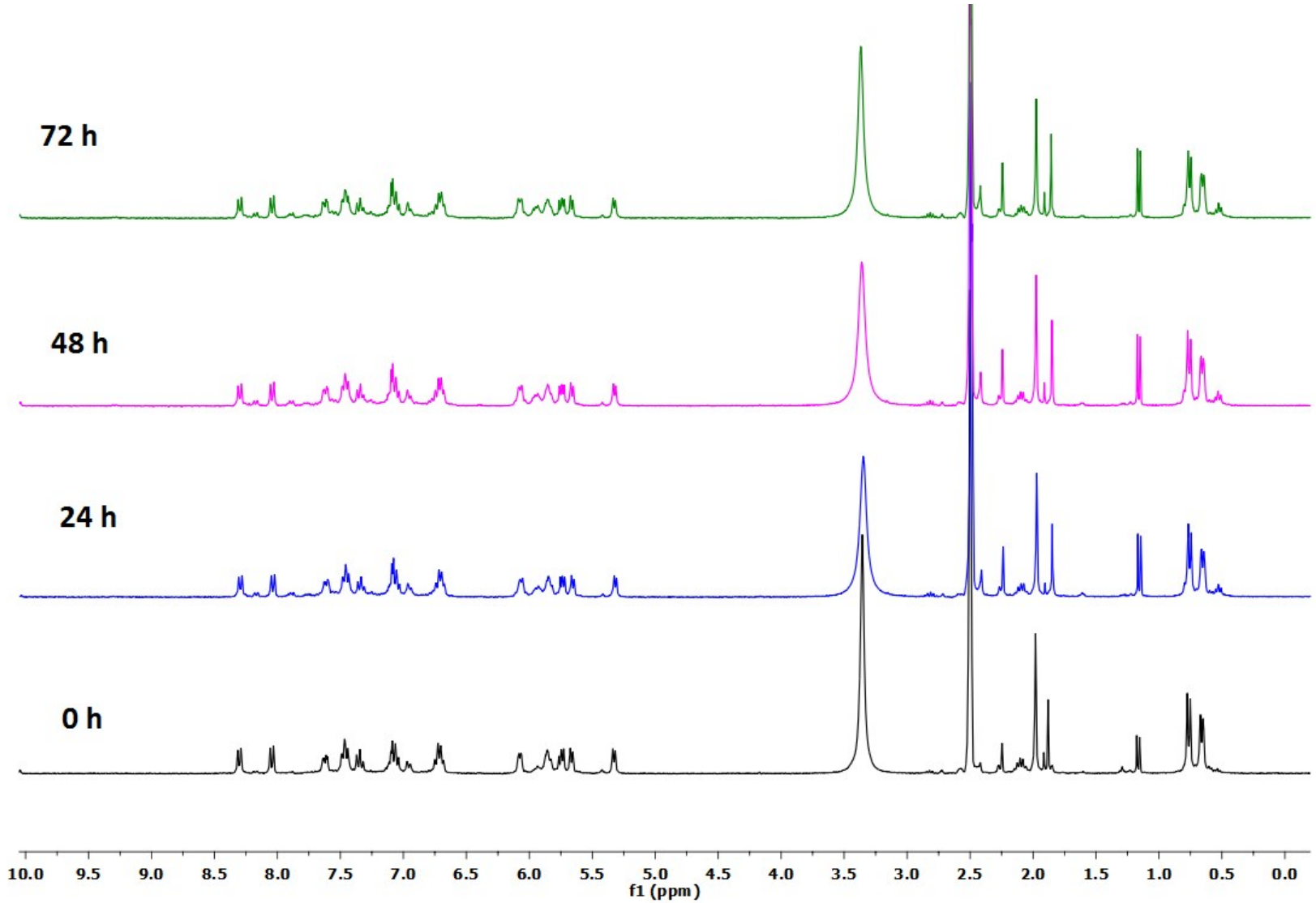


Figure S19: Time-dependent ^{1}H NMR spectra of ruthenacycle **2** in DMSO-d_6 from 0 to 72 h.

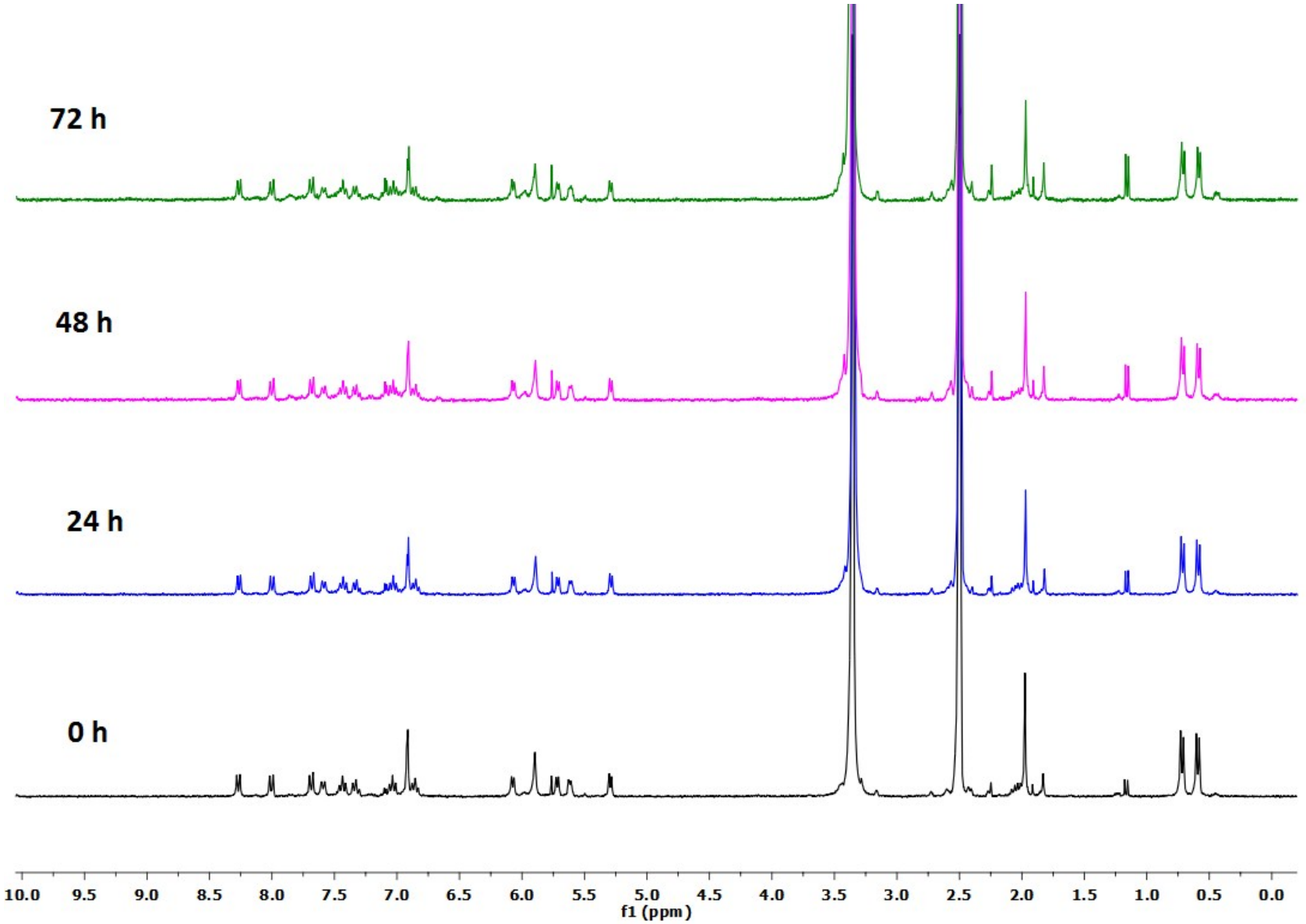


Figure S20: Time-dependent ^{1}H NMR spectra of ruthenacycle **3** in DMSO-d_6 from 0 to 72 h.

Table S1. Crystal and structure refinement data of doubly cyclometalated ruthenacycle **3**

	3
Formula	C83 H85 Cl5 N6 O2 Ru3
mol wt	1679.02
temp (K)	100(2) K
wavelength (λ)	0.70000 Å
crystsyst	Triclinic
space group	*P*-1
a (Å)	10.432(2)
b (Å)	14.840(3)
c (Å)	24.623(5)
α (deg)	80.08(3)
β(deg)	82.83(3)
γ (deg)	80.63(3)
volume (Å^3)	3686.4(14)
Z	2
ρ_{calcd} (g cm^{-3})	1.513
F(000)	1716
μ(Mo Kα) (mm^{-1})	0.797
θ range (deg)	1.502 to 32.714
no. of reflnscoll	37272
no. of reflns used	19266
R1 [I > 2σ(I)]	R_1 = 0.0687
wR2 (all reflns)	wR_2 = 0.2006
goodness of fit on F^2	1.026
largest diff peak/hole (e · Å^{-3})	1.457/-1.411