Supporting Information for

Synthesis of a chiral dinuclear Cu(II)-benzothiazolamine complex: evidence of cuprophilic interaction in its structure, exploration of its electrochemical property, and catalytic performance.

O. Stephen Ojo*, Halilu Sale, Mark D. Symes and Claire Wilson

WestCHEM, School of Chemistry, The Joseph Black Building, University of Glasgow, Glasgow, G12 8QQ, United Kingdom.

*Corresponding author

Telephone: +44 (0)141 330 6334

Email: Oluwarotimi.Ojo@glasgow.ac.uk

General Methods

Synthesis:

All reagents were obtained from commercial sources and used as received. All solvents were analytical grade and used as purchased. Brine refers to a saturated aqueous solution of sodium chloride. Flash column chromatography was performed using silica gel 60Å (35–70 μm). Merck Kieselgel aluminium-backed plates precoated with silica gel 60Å F254 were used for thin-layer chromatography (TLC) and were visualised by ultraviolet light (254 nm) and/or heating the plate after staining with vanillin or potassium permanganate ($KMnO_4$). The ^1H NMR spectra were recorded on Bruker Avance III HD 500 (500 MHz) or Avance III HD 400 (400 MHz) ultrashield™ spectrometers and data are reported as follows: chemical shift (δ_H) in ppm relative to tetramethylsilane as the internal standard ($CDCl_3$, δ 7.26 ppm; DMSO-d_6 δ 2.50 ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or overlap of non-equivalent resonances), and integration. The ^{13}C NMR spectra were recorded on NMR spectrometer at either 101 or 126 MHz and data are reported as follows: chemical shift (δ_C) in ppm relative to tetramethylsilane or the solvent as an internal standard ($CDCl_3$, δ 77.16 ppm; DMSO-d_6 δ 39.5 ppm). The multiplicity of each carbon with respect to hydrogen was deduced from DEPT (Distortionless Enhancement by Polarization Transfer) 135 experiments, which determined whether they are C, CH, CH_2, or CH_3. All the ^{13}C and ^{19}F NMR were ran and reported as hydrogen decoupled. The infrared spectra were recorded on a Perkin Elmer FT-IR spectrometer using neat samples; Wavelengths of maximum absorbance (ν_{max}) are quoted in wavenumbers (cm^{-1}). Only selected, characteristic IR absorption data are provided for each compound. The mass spectra were recorded using electron ionisation (EI) or electrospray ionisation (ESI) techniques. The parent ion $[M]^+$, $[M+H]^+$ or $[M+Na]^+$ is calculated to 4 decimal places from the molecular formula, and all values are within a tolerance of 5 ppm. Elemental analysis, mass spectrometer analysis and x-ray analysis were run by school of chemistry, University of Glasgow analytical service team.

Electrochemical measurement:

A Gamry potentiostat (Gamry Instruments, interface 1010E) was used to carry out cyclic voltammetry tests. The electrochemical measurements were undertaken using a conventional three-electrode glass cell consisting of working, counter, and reference electrodes. The working electrode was a glassy carbon (BASi, diameter = 3.0 mm) having a surface area of 0.071 cm^2, while the counter electrode was a platinum foil. A saturated calomel electrode (SCE) was used as the reference electrode, and all potentials were measured with respect to the SCE. The glassy carbon electrode was polished by applying a 0.05 μm alumina suspension (BASi) onto an alumina polishing pad to achieve a fresh surface. Subsequently, the electrode was thoroughly washed with distilled water. The cyclic voltammetry experiments were conducted in the presence of 0.01 M Cu(II) or Cu(I) complexes in CH_3OH (Honeywell, 99.7% purity), 0.1 M tetraethylammonium chloride (Sigma-Aldrich, 97.0% purity) was used as a supporting electrode, at room temperature (~25 °C) after purging the solution for 20 minutes to remove any dissolved oxygen from the system.

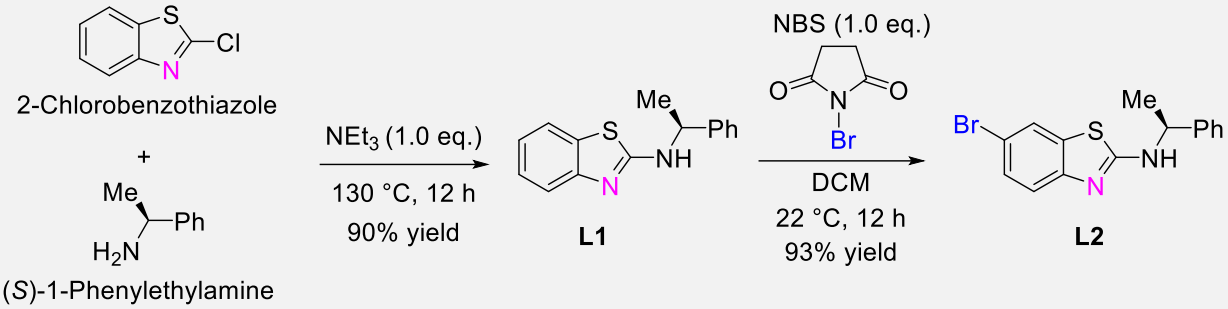
Thermal measurement (TGA and DSC):

Thermogravimetric analysis (TGA) was gathered as follows: DT Q600 , manufactured by TA Instruments; ramp from RT to 300 °C at 10°/min in 100ml/min Argon.
Differential Scanning Calorimetry (DSC) was gathered as follows: Discovery DSC 25, manufactured by TA Instruments; ramp from RT to (temperature below decomposition temperature found by TGA) at 10°/min.

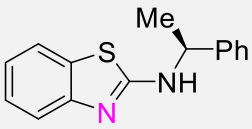
Elemental Analysis (EA):

All samples were measured on the CE-440 Elemental Analyzer by Exeter Analytical Inc., Version 10.03.21DDSA. Standard running conditions are combustion temperature (975 °C +/- 10°C), reduction temperature (620 °C +/- 20 °C), oven temperature (80 °C +/- 2 °C), purge time (60 seconds), combustion time (60 seconds), helium outlet pressure (~1.0 bar to give Fill time of 30–50 sec), oxygen outlet pressure (~1.3 bar).

Synthesis of benzothiazole-2-amines, L1 and L2

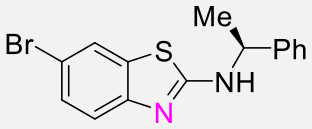


N-[(1*S*)-1-Phenylethyl]-2-benzothiazolamine (**L1**)



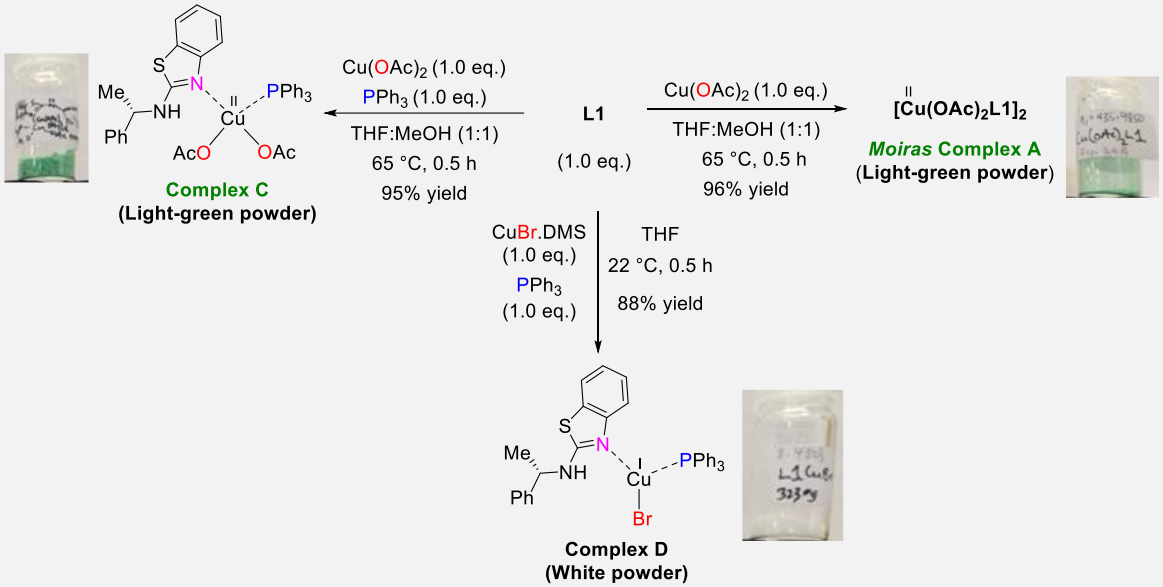
2-chlorobenzothiazole (2.30 mL, 3.00 g, 17.6855 mmol, 1.0 eq.) was added to a stirring mixture of (*S*)-1-phenylethylamine (2.14 g, 17.6855 mmol, 1.0 eq.) and NEt_3 (2.28 mL, 1.79 g, 17.6855 mmol, 1.0 eq.) in a 50 mL round bottom flask. The resulting mixture was heated to 130 ℃ and left to stir at this temperature for 12 h. Afterwards, the viscous mixture was allowed to cool to 40 ˚C then immediately diluted with saturated aqueous solution of ammonium chloride (100 mL) and extracted with DCM (60 mL) twice. The combined organic layers were washed with brine (30 mL), dried with $MgSO_4$, filtered and solvent removed *in vacuo*. The crude sample was recrystallised in hexane. Vacuum filtration provided **L1** (4.05 g, 15.9169 mmol, 90%) as a white crystalline solid. NEt_3 = Triethylamine. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.52 (d, *J* = 7.7 Hz, 1H, *N*-H), 7.64 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.39–7.42 (m, 2H), 7.31–7.35 (m, 3H), 7.16–7.25 (m, 2H), 6.99 (d, *J* = 7.7, 1.3 Hz, 1H), 5.04 (p, *J* = 7.0 Hz, 1H), 1.49 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (101 MHz, DMSO-*d6*) δ 165.3, 152.4, 144.4, 130.2, 128.3, 126.8, 125.9, 125.4, 120.8, 118.0, 53.3, 23.1. **IR** (neat) V_{max} (cm^{-1}): 2966, 1601, 1568, 1552, 1443. **HRMS** (ESI): calc for $C_{15}H_{14}N_2S$ *m/z*: 254.0878 found $[M+H]^+$ 255.0956. **MP**: 130-132 ℃.

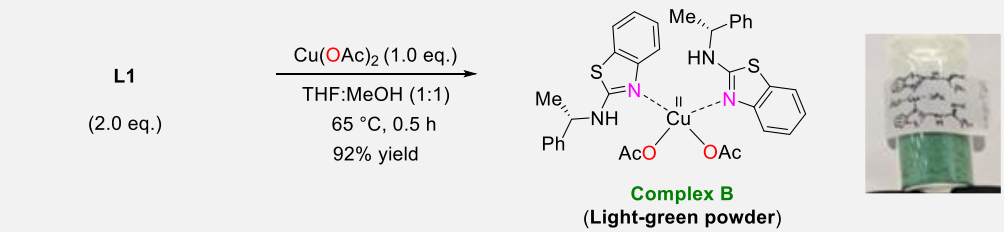
6-Bromo-*N*-[(1*S*)-1-phenylethyl]-2-benzothiazolamine (**L2**)

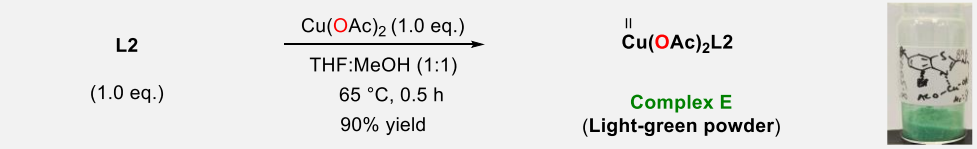


NBS (1.34 g, 7.5521 mmol, 1.0 eq.) was added to a stirring solution of (*S*)-**L1** (1.92 g, 7.5521 mmol, 1.0 eq.) in DCM (30 mL) at room temperature and left to stir for 12 h (the mixture immediately turned red, then after 1 h turned light brown). Afterwards, the mixture was washed with saturated aqueous solution of sodium hydrogen carbonate (35 mL) and the aqueous layer was further extracted with DCM (40 mL). The combined organic layers were washed with brine (35 mL), dried with $MgSO_4$, filtered and solvent removed *in vacuo* to generate (*S*)-**L2** (2.35 g, 7.049 mmol, 93%) as a brown solid. NBS = *N*-bromosuccinimide, DCM = Dichloromethane. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.65 (d, *J* = 7.7 Hz, 1H, *N*-H), 7.89 (s, 1H), 7.38–7.41 (m, 2H), 7.30–7.35 (m, 3H), 7.21–7.26 (m, 2H), 5.03 (p, *J* = 7.0 Hz, 1H), 1.48 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (101 MHz, DMSO-*d6*) δ 165.8, 151.6, 144.1, 132.4, 128.39, 128.34, 126.9, 125.9, 123.3, 119.4, 112.1, 53.4, 23.0. **IR** (neat) V_{max} (cm^{-1}): 2969, 1594, 1555, 1443, 1254. **HRMS** (ESI): calc for $C_{15}H_{13}N_2S^{79}Br$ *m/z*: 331.9983 found $[M+H]^+$ 333.0061. **MP**: 120-122 ℃.

Scheme S1: Synthesis of Complexes A-E







tris(μ-acetato)-(μ-acetato)-bis[*N*-(1-phenylethyl)-1,3-benzothiazol-2-amine]-di-copper – Moiras Complex A

A colourless solution of (*S*)-**L1** (0.21 g, 0.8259 mmol, 1.0 eq.) in THF (5 mL) was added to a stirring deep blue solution of $Cu(OAc)_2$ (0.15 g, 0.8259 mmol, 1.0 eq.) in MeOH (5 mL) at room temperature. The resulting reaction mixture was heated to 65 °C and left to stir at this temperature for 0.5 h. Afterwards, the mixture was allowed to cool to room temperature, and solvent was removed *in vacuo*. The resulting light-green powder was washed with hexane and collected by vacuum filtration. **Moiras complex A** (0.35 g, 0.7929 mmol, 96%) was obtained as a light-green powder. MeOH = Methanol. **IR** (neat) V_{max} (cm^{-1}): 1621, 1596, 1571, 1556, 1454, 1421. **Elemental Analysis**: cal. C, 52.34%; H, 4.62%; N, 6.43%; O, 14.68%; S, 7.35%, Cu, 14.58%. Observ. C, 52.34%; H, 4.62%; N, 6.43%; O, 14.68%; S, 7.35%, Cu, 14.58%.

Complex B – $Cu(OAc)_2(L1)_2$

A colourless solution of (*S*)-**L1** (0.42 g, 1.6518 mmol, 2.0 eq.) in THF (5 mL) was added to a stirring deep blue solution of $Cu(OAc)_2$ (0.15 g, 0.8259 mmol, 1.0 eq.) in MeOH (5 mL) at room temperature. The resulting reaction mixture was heated to 65 °C and left to stir at this temperature for 0.5 h. Afterwards, the mixture was allowed to cool to room temperature, and solvent was removed *in vacuo*. The resulting green powder was washed with hexane and collected by vacuum filtration. **Complex B** (0.52 g , 0.7598 mmol, 92%) was obtained as a light-green powder (0.52 g , 0.7598 mmol, 92%). **IR** (neat) V_{max} (cm^{-1}): 1624, 1596, 1557, 1453, 1424. **Elemental Analysis**: cal. C, 59.16%; H, 4.96%; N, 8.12%; O, 9.27%; S, 9.29%; Cu, 9.21%. Observ. C, 59.16%; H, 4.96%; N, 8.12%; O, 9.27%; S, 9.29%, Cu, 9.21%.

Complex C – $Cu(OAc)_2L1PPh_3$

$Cu(OAc)_2$ (0.15 g, 0.8259 mmol, 1.0 eq.) was added, all at once, to a stirring colourless solution of (*S*)-**L1** (0.21 g, 0.8259 mmol, 1.0 eq.) and PPh_3 (0.22 g, 0.8259 mmol, 1.0 eq.) in THF (5 mL) and MeOH (5 mL) at room temperature. The resulting reaction mixture was heated to 65°C and left to stir at this temperature for 0.5 h. Afterwards, the mixture was allowed to cool to room temperature, and solvent was removed *in vacuo*. The resulting green powder was washed with hexane and collected by vacuum filtration. **Complex C** (0.55 g, 0.7846 mmol, 95%) was obtained as a light-green powder. PPh_3 = Triphenylphosphine. **^{31}P NMR** (162 MHz, $CDCl_3$): δ -2.65. **IR** (neat) V_{max} (cm^{-1}): 1624, 1596, 1558, 1452, 1420. **Elemental Analysis**: calc. C, 63.64%; H, 5.05%; N, 4.01%; O, 9.16%, P, 4.44%; S, 4.59%; Cu, 9.1%. Observ. C, 63.64%; H, 5.05%; N, 4.01%; O, 9.16%; P, 4.44%; S, 4.59%; Cu, 9.1%.

Complex D – $CuBrL1PPh_3$

CuBr.DMS (0.40 g, 1.9656 mmol, 1.0 eq.) was added, all at once, to a stirring colourless solution of (*S*)-**L1** (0.50 g, 1.9656 mmol, 1.0 eq.) and PPh_3 (0.52 g, 1.9656 mmol, 1.0 eq.) in THF (15 mL) at room temperature left to stir for 0.5 h. Afterwards, the mixture was allowed to cool to room temperature, and solvent was removed *in vacuo*. The resulting white fluffy powder was rinsed with hexane and collected by vacuum filtration. **Complex D** (1.14 g, 1.7297 mmol, 88%) was obtained as a white solid. PPh_3 = Triphenylphosphine. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.83 (s, 1H, *N*-H), 7.53–7.57 (m, 6H), 7.42–7.44 (m, 3H), 7.34–7.39 (m, 4H), 7.26–7.33 (m, 9H), 6.94–7.02 (m, 2H), 4.49 (q, *J* = 6.8 Hz, 1H), 4.49 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.0, 150.1, 142.6, 134.2, 134.1, 133.0, 132.6, 129.9, 128.9, 128.7, 128.6, 127.7, 126.4, 126.0, 121.8, 120.8, 119.3, 57.0, 24.4. **^{31}P NMR** (162 MHz, $CDCl_3$): δ -4.60. **IR** (neat) V_{max} (cm^{-1}): 1548, 1449, 1432, 1092. **HRMS** (ESI): calc for $C_{33}H_{29}N_2Cu^{79}BrPS$ *m/z*: 658.0268 found $[M+H]^+$ 659.0346. **Elemental Analysis**: cal. C, 60.05%; H, 4.43%; N, 4.24%; Br, 12.10%; P, 4.69%; S, 4.86%, Cu, 9.63%. Observ. C, 60.05%; H, 4.43%; N, 4.24%; Br, 12.10%; P, 4.69%; S, 4.86%, Cu, 9.63%.

Complex E – $Cu(OAc)_2$L2

A colourless solution of (*S*)-**L2** (0.28 g, 0.8259 mmol, 1.0 eq.) in THF (5 mL) was added to a stirring deep blue solution of $Cu(OAc)_2$ (0.15 g, 0.8259 mmol, 1.0 eq.) in MeOH (5 mL) at room temperature. The resulting reaction mixture was heated to 65 °C and left to stir at this temperature for 0.5 h. Afterwards, the mixture was allowed to cool to room temperature, and solvent was removed *in vacuo*. The resulting green powder was washed with hexane and collected by vacuum filtration. **Complex E** (0.38 g , 0.7433 mmol, 90%) was obtained as a light-green powder. **IR** (neat) V_{max} (cm^{-1}): 1622, 1593, 1561, 1549, 1450, 1420. **Elemental Analysis**: cal. C, 44.32%; H, 3.72%; N, 5.44%; Br, 15.52%; O, 12.43%; S, 6.23%, Cu, 12.34%. Observ. C, 44.32%; H, 3.72%; N, 5.44%; Br, 15.52%; O, 12.43%; S, 6.23%, Cu, 12.34%.

Solvent coordination effect: Tetrahydrofuran (THF) *vs* Toluene (PhMe)

The behaviour of these copper complexes in THF and PhMe suggests that THF (polar coordinating solvent) is included in the coordination sphere of the Cu^{I} or Cu^{II} species.[1a] The colour of the Cu^{II} complexes is green, in PhMe the colour stayed green but changed to blue in THF. The Cu^{I} complex is white, in PhMe it stayed colourless but changed to light-gold in THF. The choice of solvent can play a crucial role in the success of some catalytic organic reaction.[1b] Also, a previous study has shown that solvent interaction and/or binding played a key role in the dynamic equilibrium between copper isomeric forms.[1c]

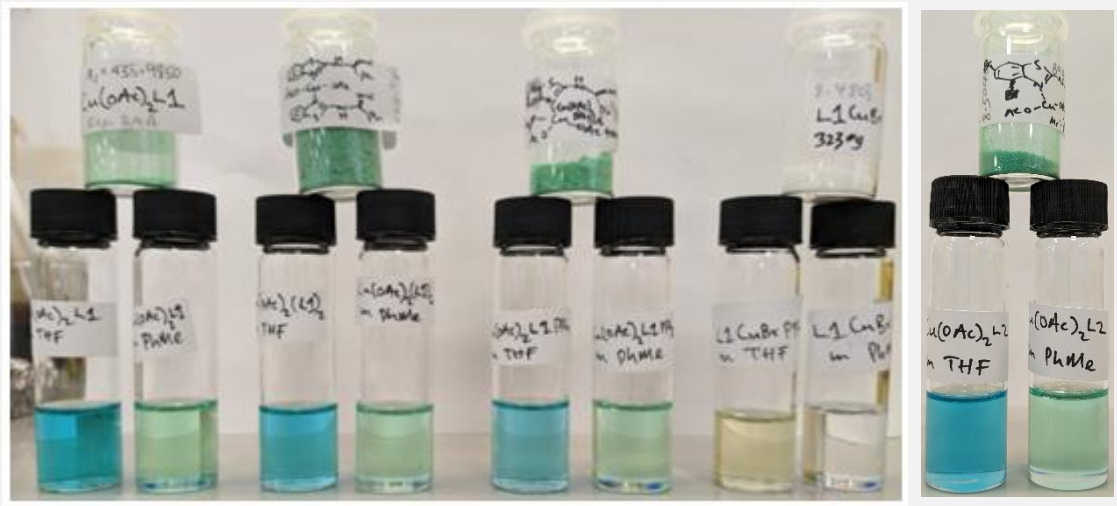


Figure S1: Behaviour of Copper complexes in solution

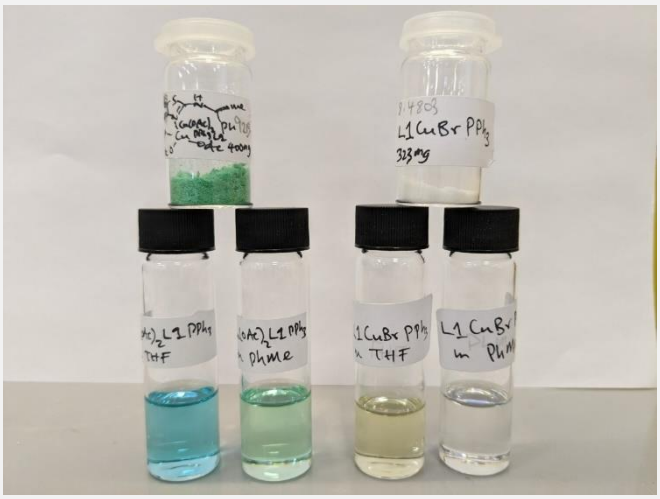


Figure S2: Cu^{II} *vs* Cu^{I}

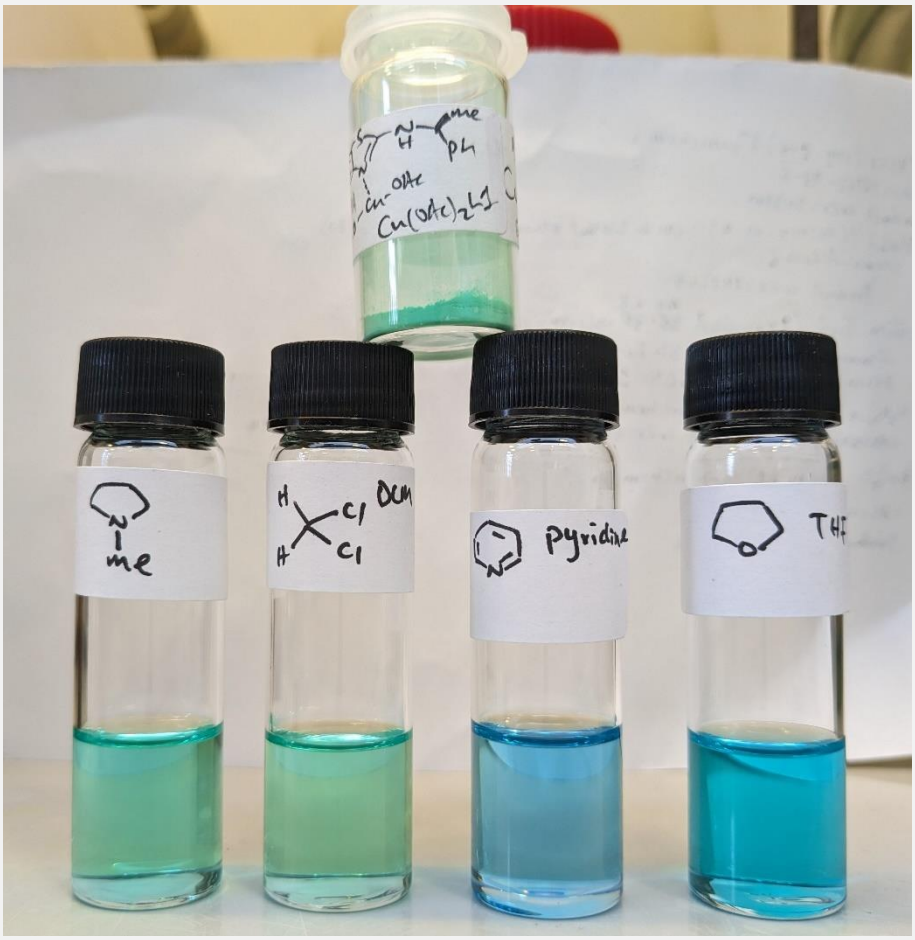


Figure S3: Complex **A** (10 mg) in 5 mL of solvent (1-Methylpyrrolidine, DCM, Pyridine or THF)

Is cuprophilic interaction between Cu(II)-Cu(II) a δ-bond?

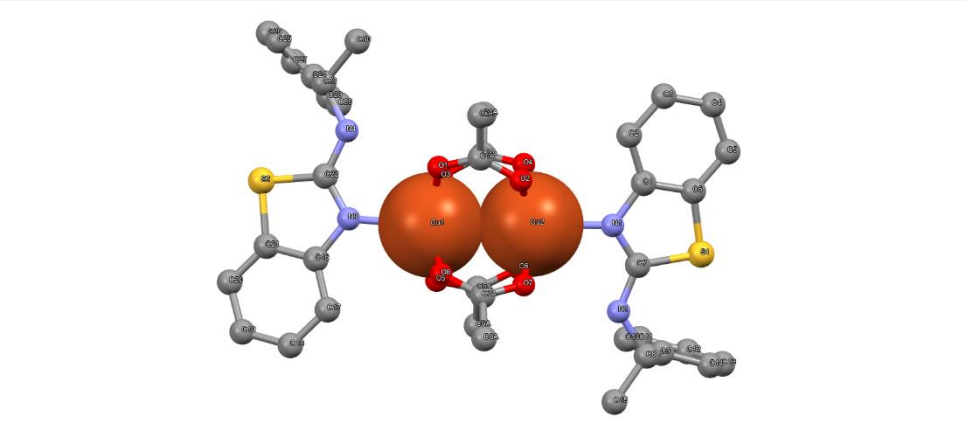
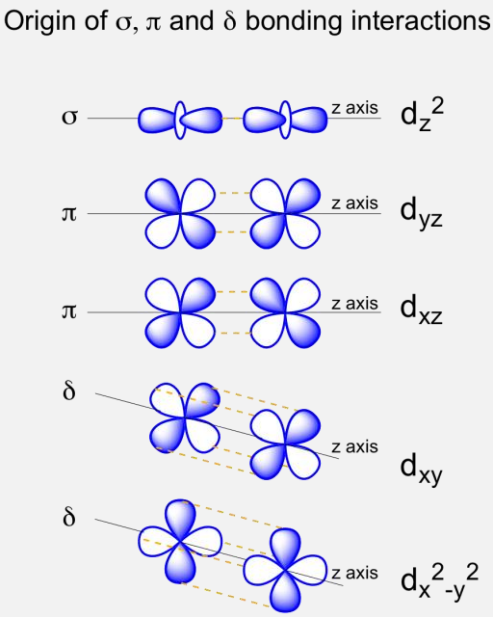
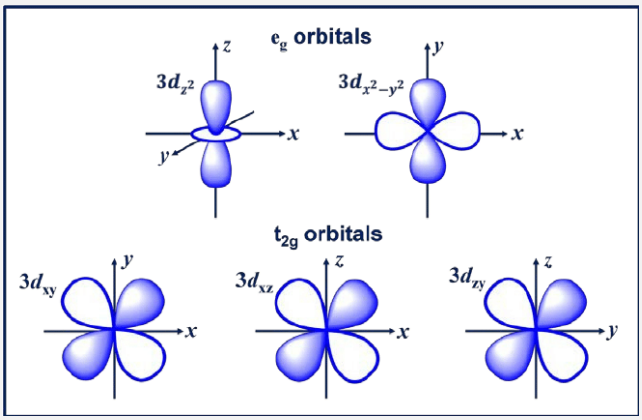


Figure S4: Two Cu(II) van der Waals radii in contact. Using Mercury software

When two spheres overlap with one another, it's an indication that the corresponding atoms are covalently bonded. When they are merely touching one another, their distance of separation is usually equal to the sum of their van der Waals (vdw) radii. In complex **A**, the chiral benzothiazolamine ligand makes this distance shorter than the sum of two Cu vdw radii. Cuprophilic interaction between Cu(I)-Cu(I) can't be a metal-metal δ-bond because of full d orbitals, $3d^{10}$-$3d^{10}$. However, Cu(II)-Cu(II) $3d^9$-$3d^9$ interaction as a δ-bond warranty an investigation since $3d_{z^2}$ (or $3d_{x2-y2}$) orbitals is partially filled.



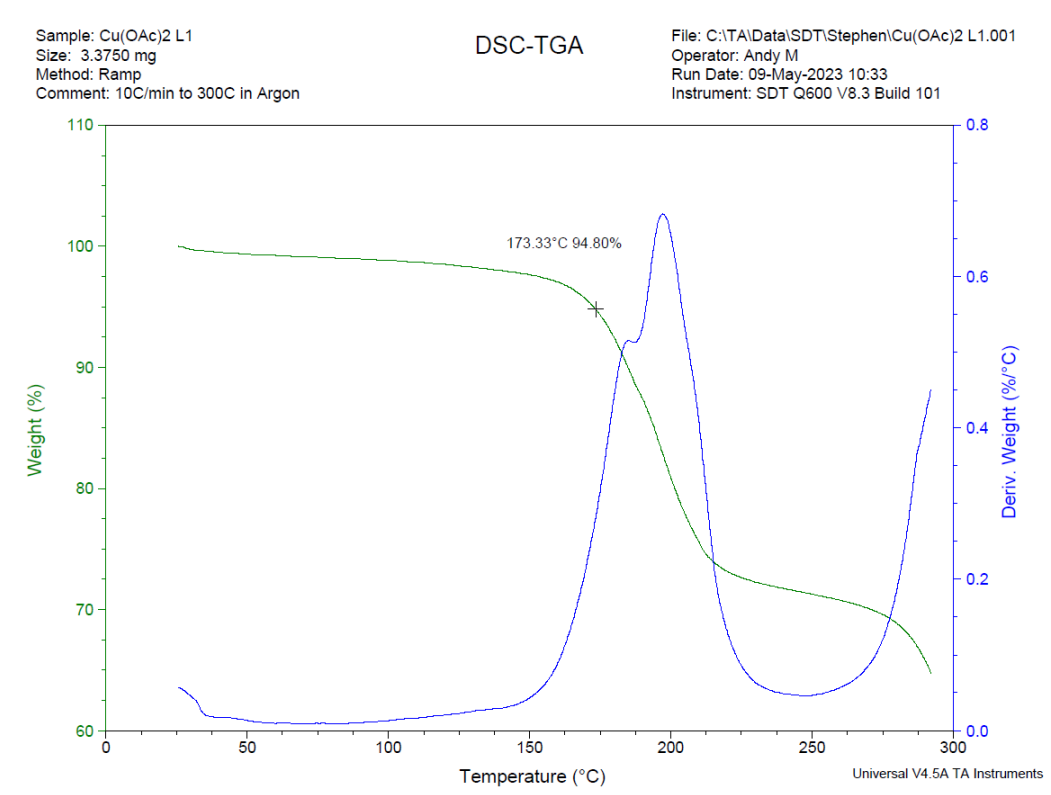


Thermal Properties of Complexes A-C and E

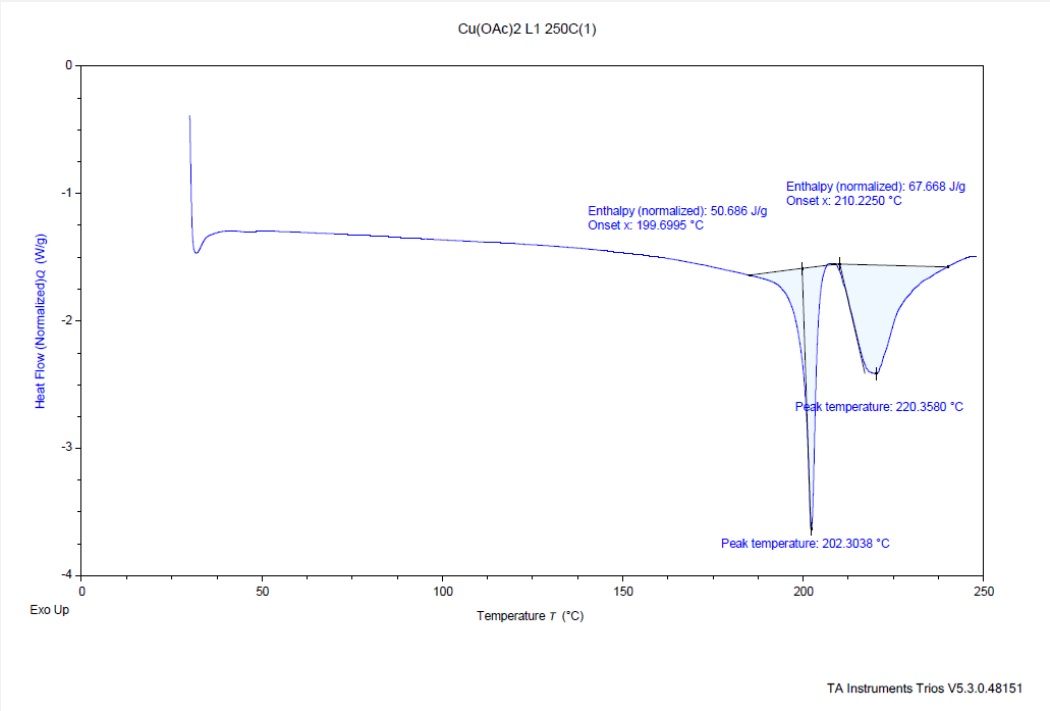
TGA and DSC Data

Moiras Complex A – $[Cu(OAc)_2L1]_2$

TGA

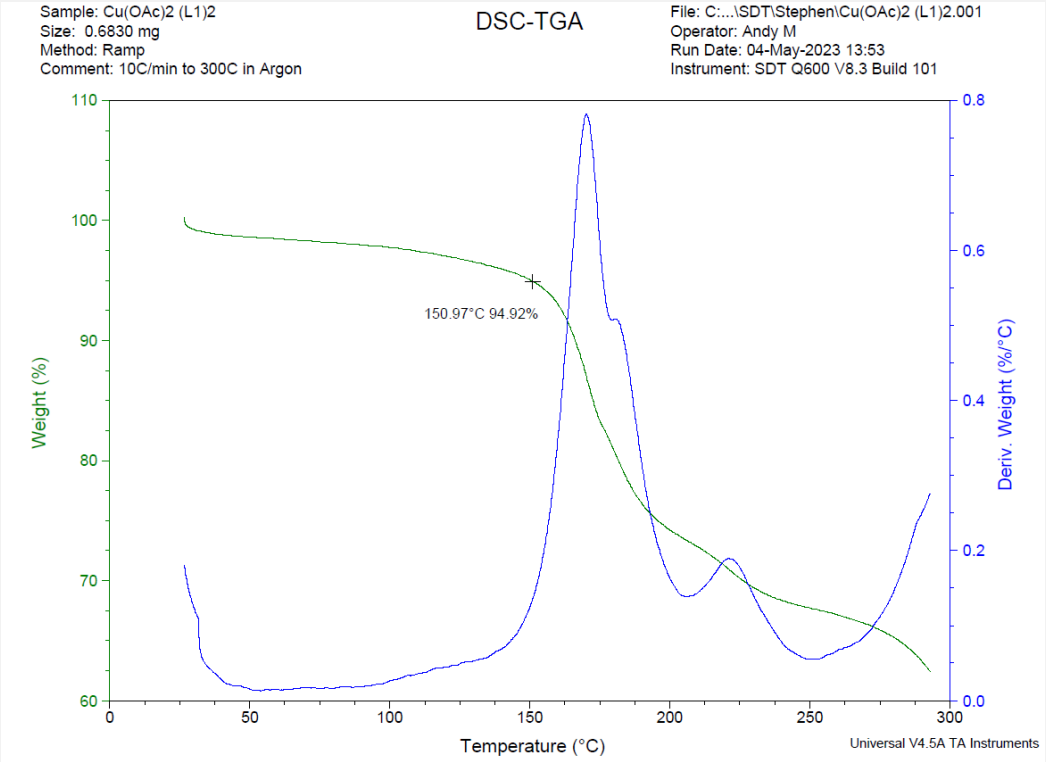


DSC

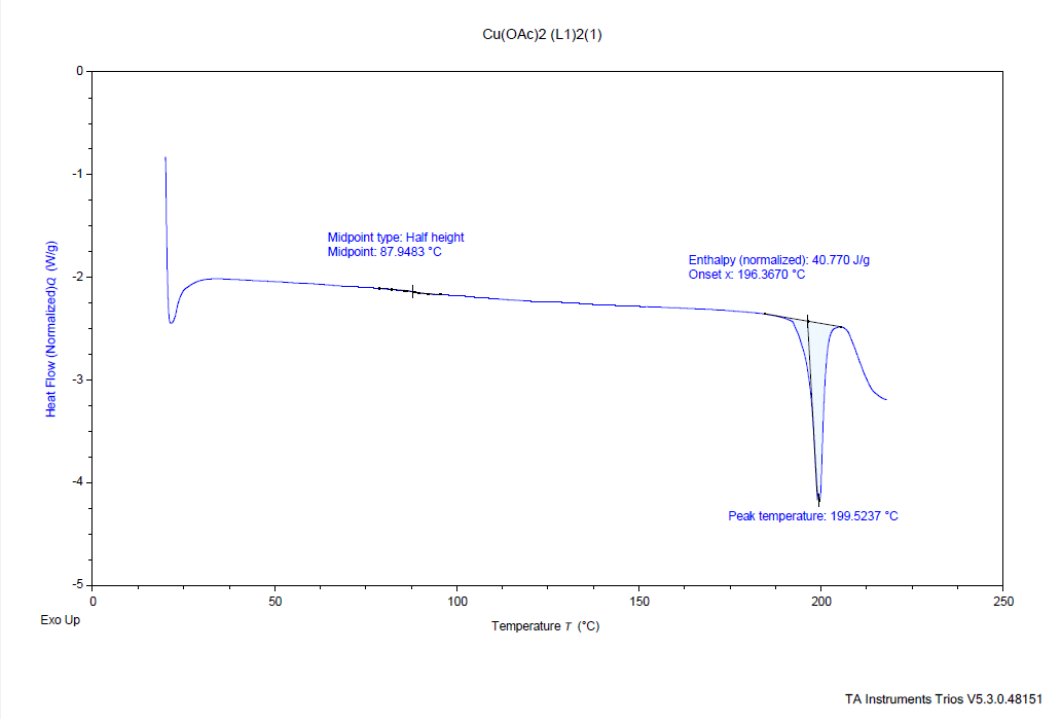


Complex B – $Cu(OAc)_2(L1)_2$

TGA

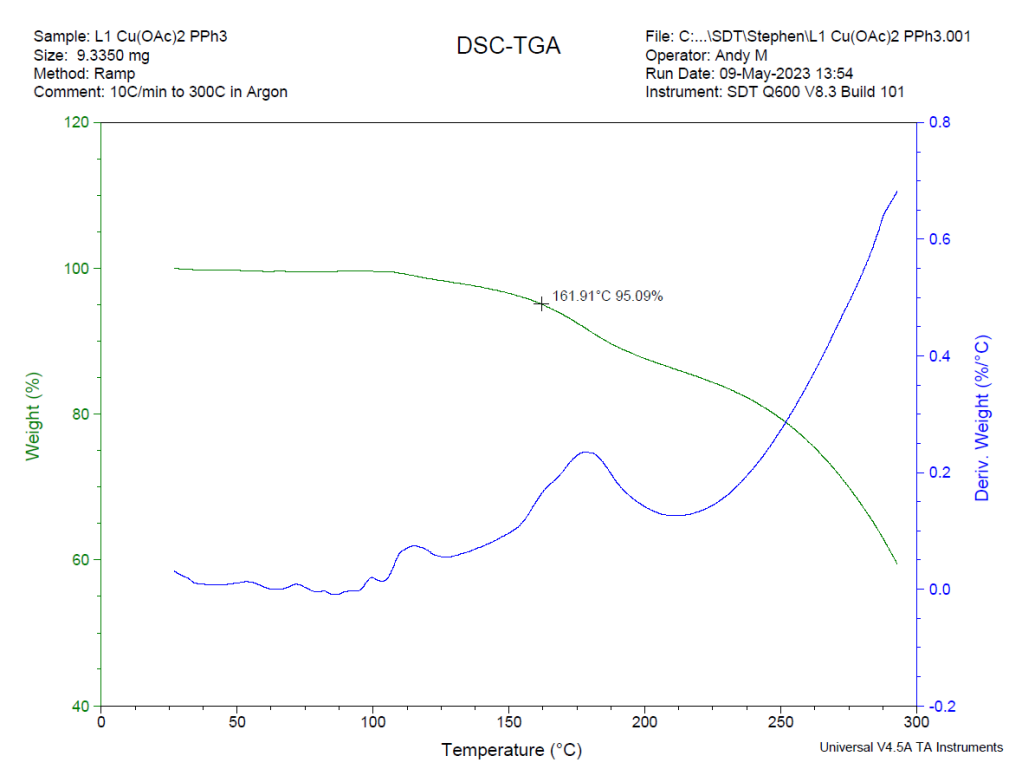


DSC

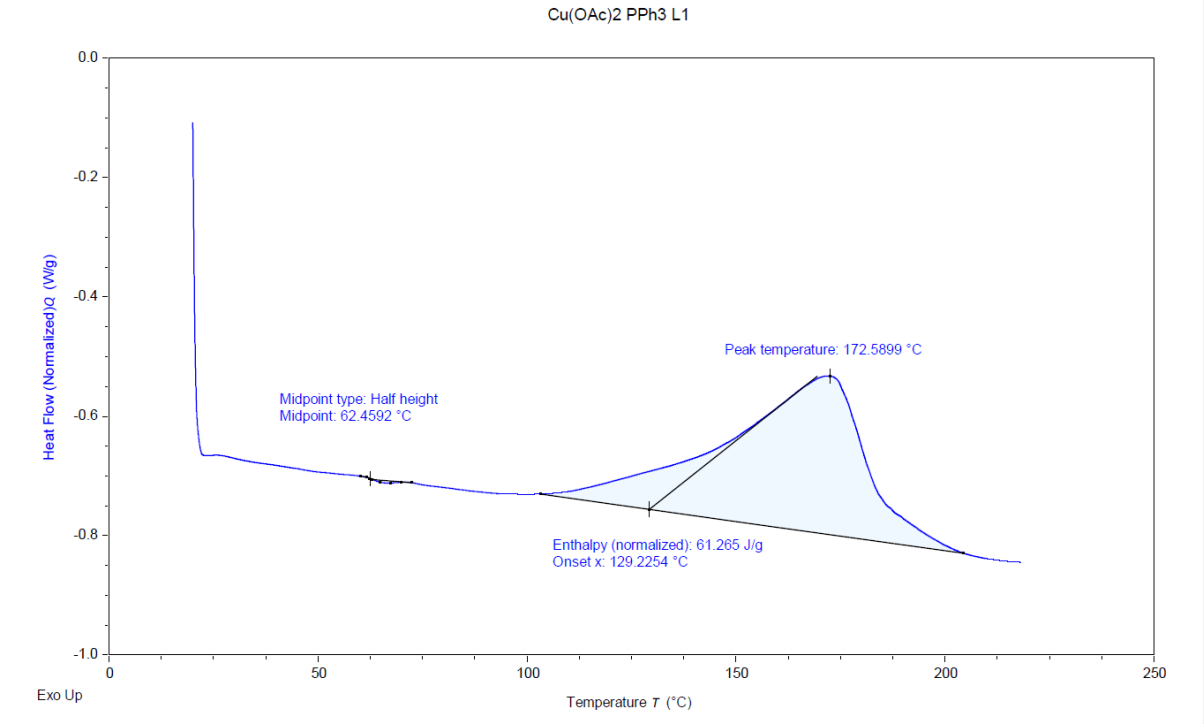


Complex C – $Cu(OAc)_2L1PPh_3$

TGA



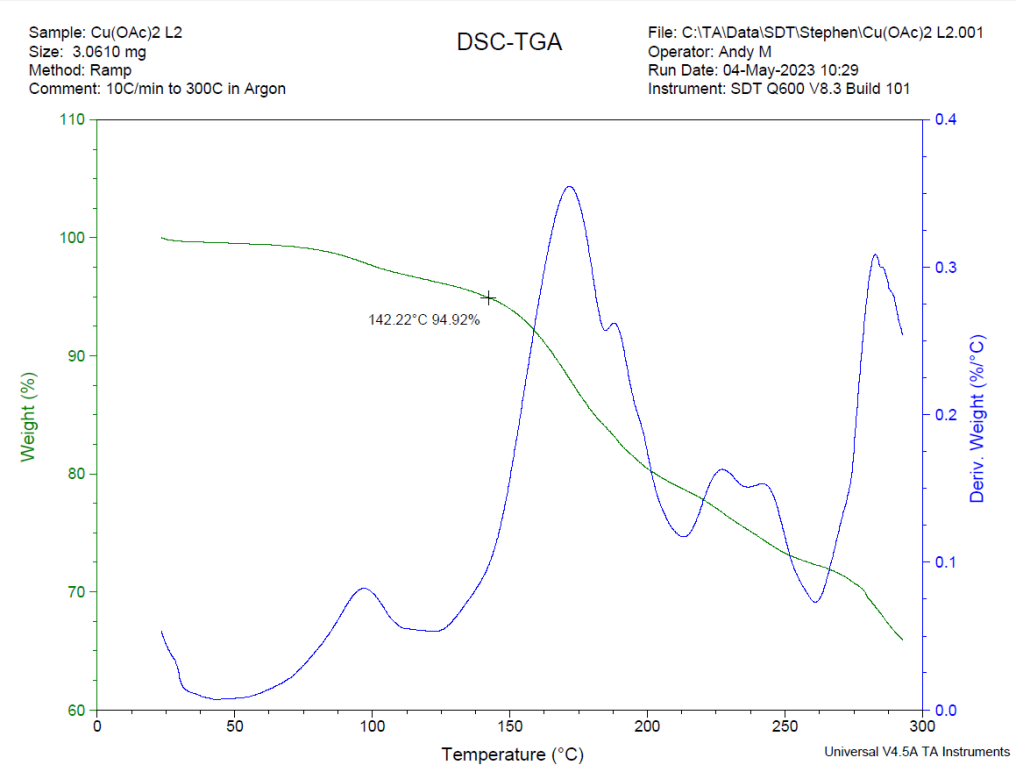
DSC



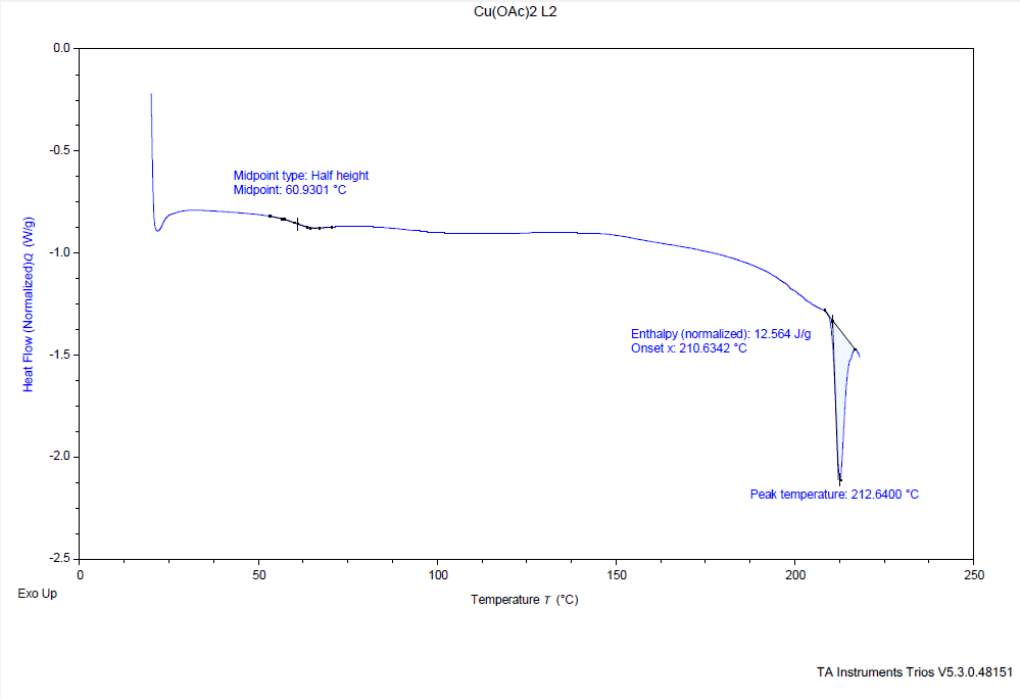
Peak at 172.5 C – indicative of exothermic reaction caused by crystallisation.

Complex E – $Cu(OAc)_2L2$

TGA

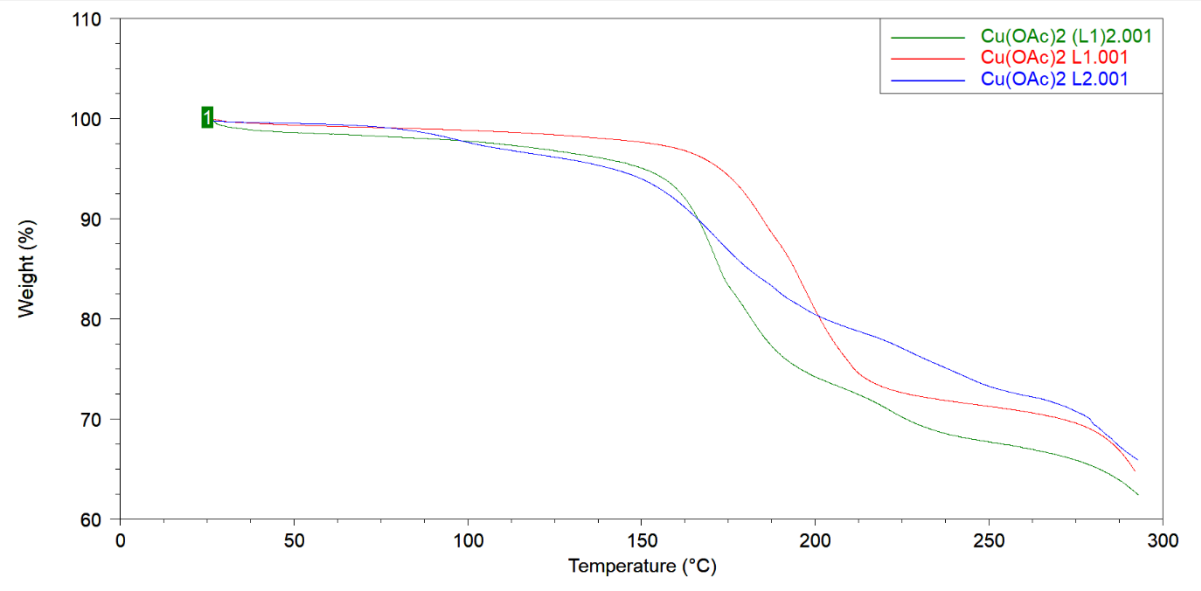


DSC

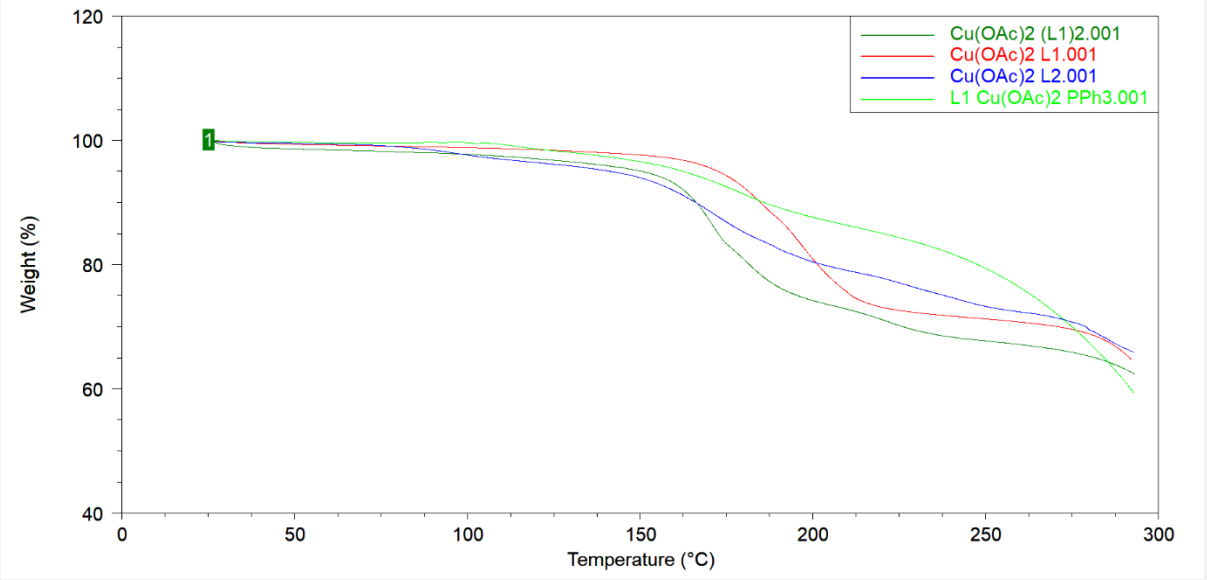


Thermogravimetric analysis:

Complex **A** *vs* **B** *vs* **E**



Complex **A** *vs* **B** *vs* **C** *vs* **E**



Electrochemical Properties of Complexes A-E

Moiras Complex A – $[Cu(OAc)_2L1]_2$

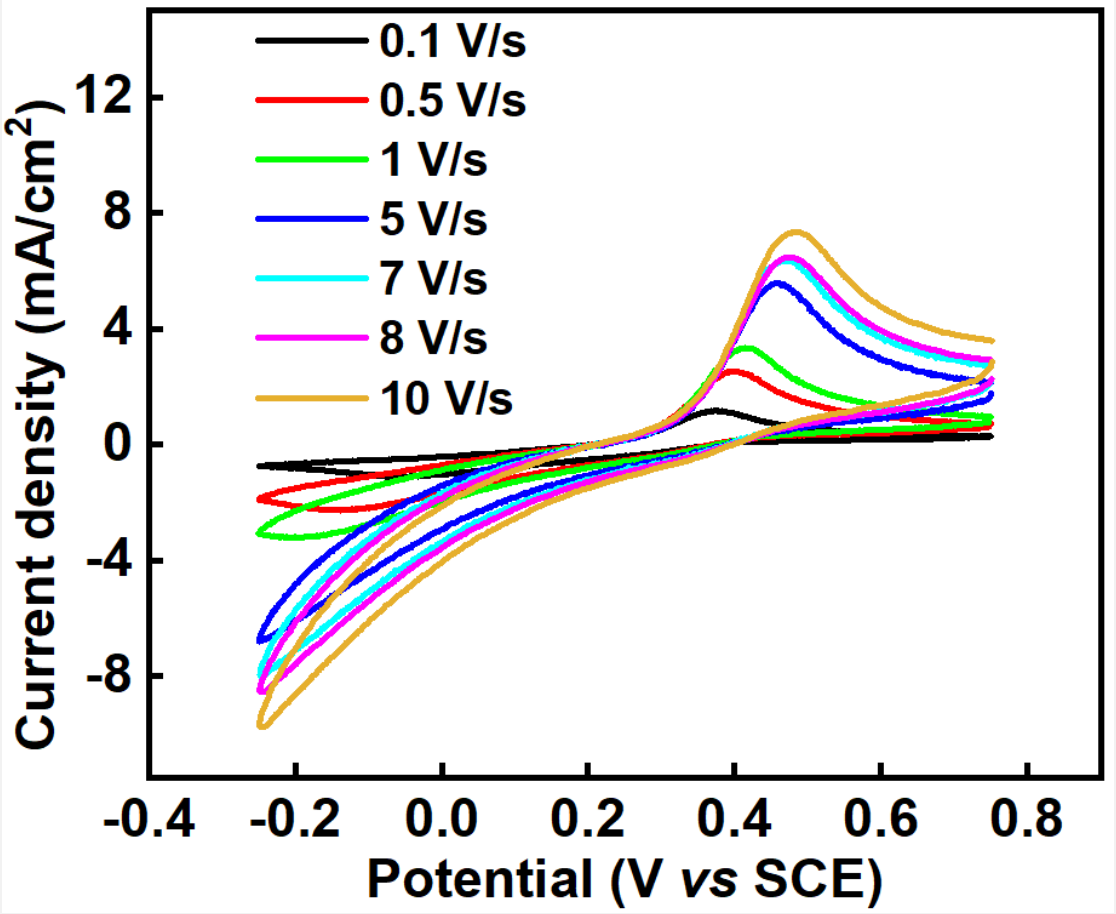


Figure S5: Cyclic voltammograms of 0.01 M $[Cu(OAc)_2L1]_2$ in CH_3OH (with 0.1 M C_8H_2OClN supporting electrolyte), glassy carbon working electrode, SCE (reference electrode), and platinum foil (counter electrode) at different scan rates (0.1 – 10 V s^{-1}).

Complex B – $Cu(OAc)_2(L1)_2$

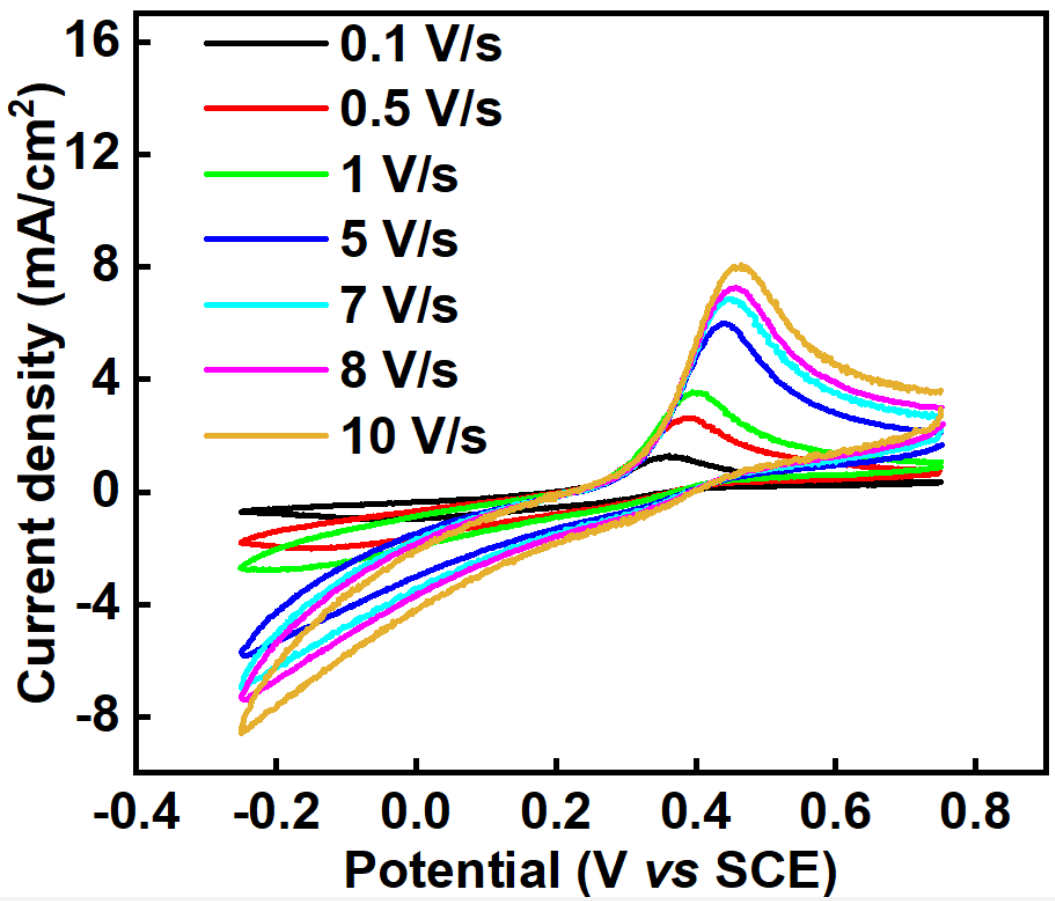


Figure S6: Cyclic voltammograms of 0.01 M $Cu(OAc)_2(L1)_2$ in CH_3OH (with 0.1 M C_8H_2OClN supporting electrolyte), glassy carbon working electrode, SCE (reference electrode), and platinum foil (counter electrode) at different scan rates (0.1 – 10 V s^{-1}).

Complex C – $Cu(OAc)_2(L1)PPh_3$

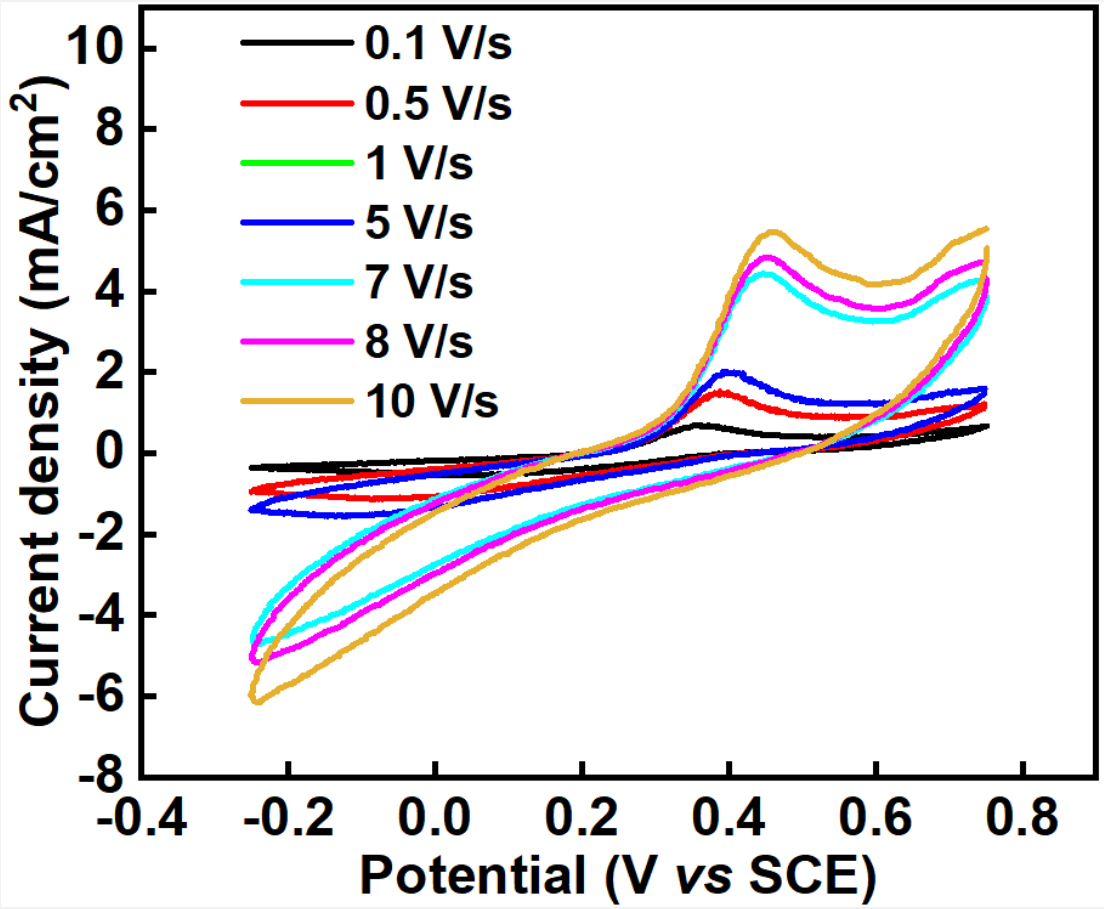


Figure S7: Cyclic voltammograms of 0.01 M $Cu(OAc)_2L1PPh_3$ in CH_3OH (with 0.1 M C_8H_2OClN supporting electrolyte), glassy carbon working electrode, SCE (reference electrode), and platinum foil (counter electrode) at different scan rates (0.1 – 10 V s^{-1}).

Complex D – CuBr(L1)PPh$_3$

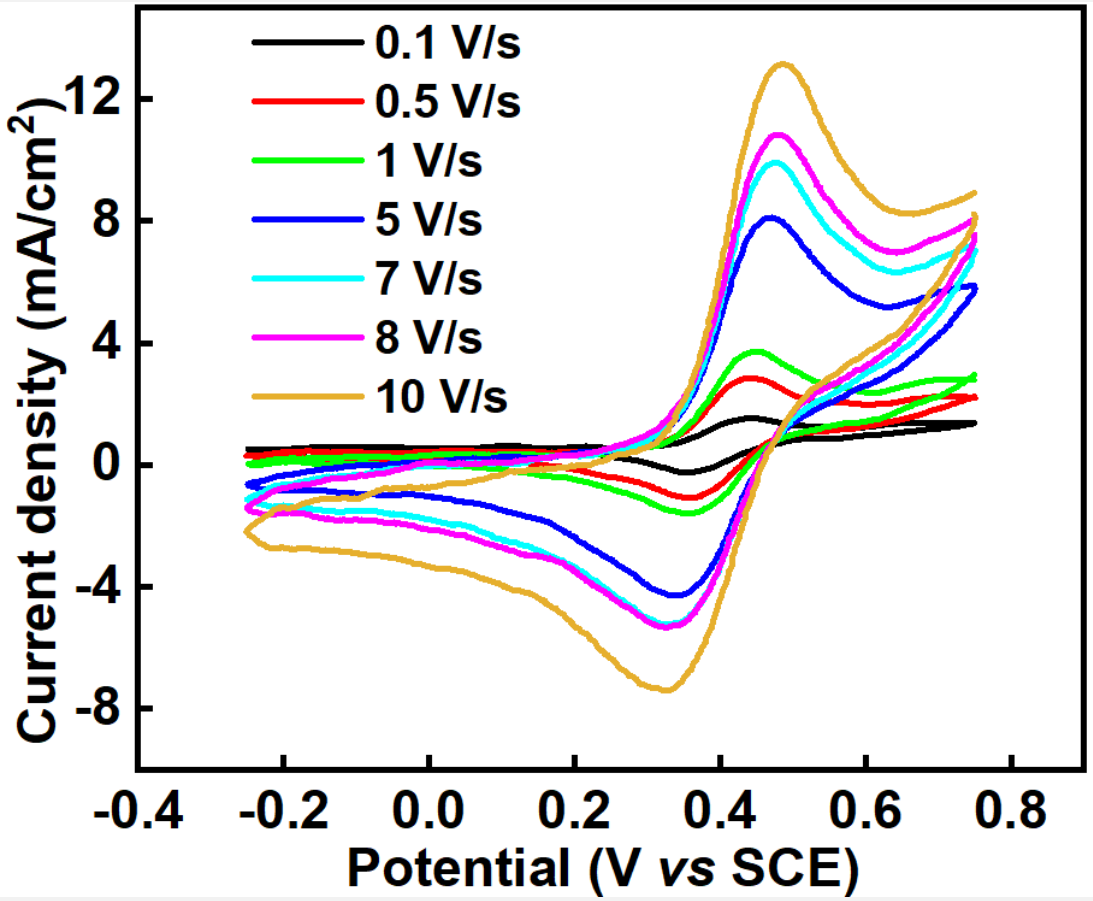


Figure S8: Cyclic voltammograms of 0.01 M CuBrL1PPh$_3$ in CH$_3$OH (with 0.1 M C$_8$H$_2$OClN supporting electrolyte), glassy carbon working electrode, SCE (reference electrode), and platinum foil (counter electrode) at different scan rates (0.1 – 10 V s^{-1}).

Complex E – $Cu(OAc)_2L2$

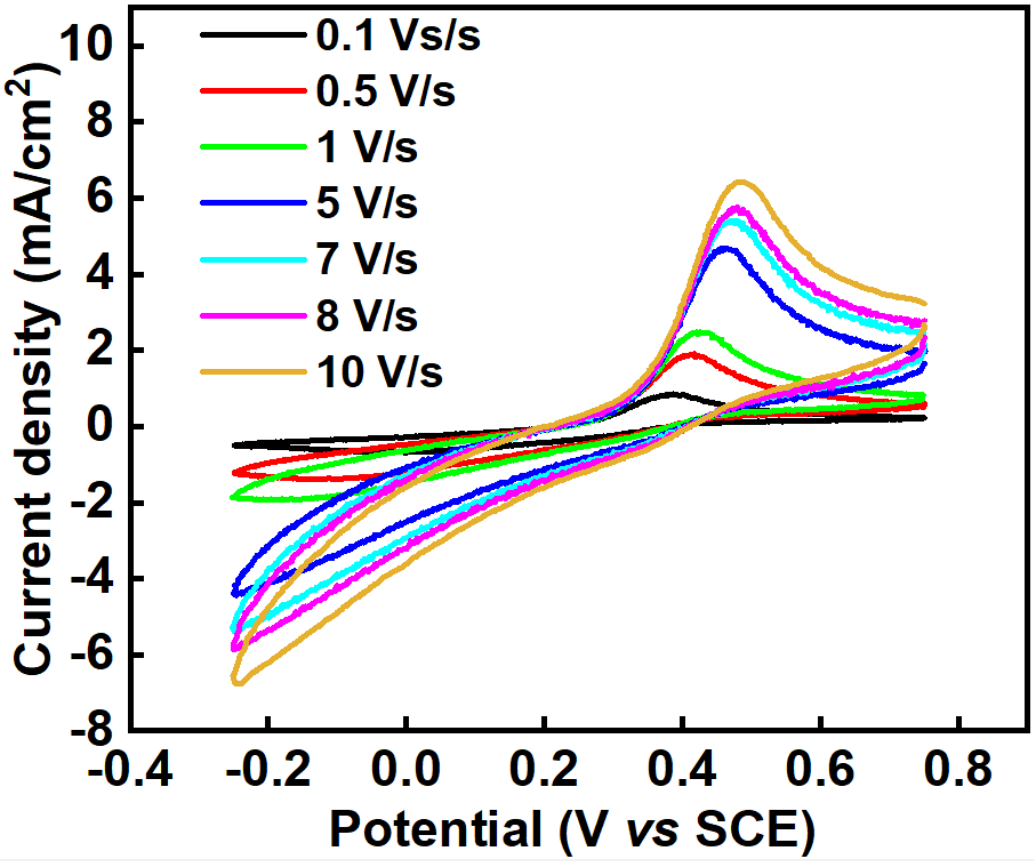
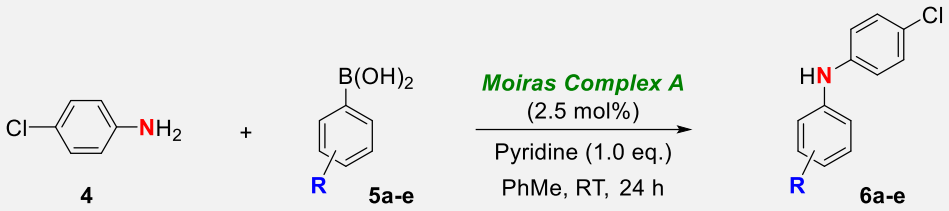


Figure S9: Cyclic voltammograms of 0.01 M $Cu(OAc)_2L2$ in CH_3OH (with 0.1 M C_8H_2OIN supporting electrolyte), glassy carbon working electrode, SCE (reference electrode), and platinum foil (counter electrode) at different scan rates (0.1 – 10 V s^{-1}).

Application of a Complex A as a catalyst in Chan-Lam reaction.

Coupling of 4-chloroaniline with arylboronic acids: C-N bond formation

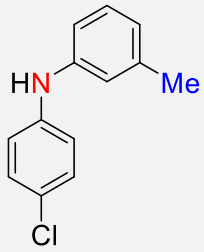


	R	Yield
6a	3-Me	92
6b	3-F	90
6c	4-F	87
6d	4-OMe	85
6e	Napthyl	89

General experimental procedure for the synthesis of 6a-e:

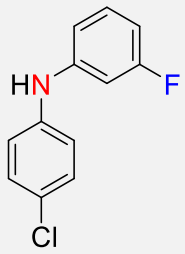
Arylamine **4** (0.20 g, 1.5677 mmol, 1.0 eq.), arylboronic acid **5** (2.3516 mmol, 1.5 eq.), and pyridine (0.12 g, 1.5677 mmol, 1.0 eq.) were dissolved in PhMe (5 mL) in a reaction vessel, then **Moiras complex A** (17.1 mg, 0.0392 mmol, 0.025 eq.) was added. The resulting mixture turned blue, and it was left to stir at room temperature for 24 h. Afterwards, it was diluted with ethyl acetate (30 mL), washed with 1 M HCl (15 mL) followed by brine (20 mL). The organic layer was dried with $MgSO_4$, filtered and solvent removed *in vacuo*. The crude mixture was purified by silica gel chromatography using 2-5% ethyl acetate in Hexanes.

***N*-(4-Chlorophenyl)-3-methylbenzenamine (6a)**



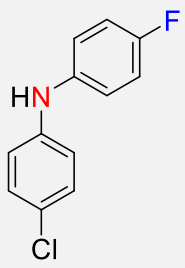
The general experimental procedure described for the synthesis of **6a-6e** was followed using 4-chloroaniline **4** (0.20 g, 1.5677 mmol, 1.0 eq.), 3-tolylboronic acid **5a** (0.32 g, 2.3516 mmol, 1.5 eq.), pyridine (0.12 g, 1.5677 mmol, 1.0 eq.), **Moiras complex A** (17.1 mg, 0.0392 mmol, 0.025 eq.) and PhMe (5 mL). Compound **6a** was obtained as a brown oil (0.31 g, 1.4423 mmol, 92%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.21 (d, *J* = 8.9 Hz, 2H), 7.14–7.19 (m, 1H), 6.98 (d, *J* = 8.9 Hz, 2H), 6.84–6.89 (m, 2H), 6.78 (d, *J* = 7.7 Hz, 1H), 5.62 (s, 1H, *N*-H), 2.32 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.7, 142.1, 139.5, 129.4, 129.3, 125.5, 122.5, 118.9, 115.3, 21.6. **IR** (neat) V_{max} (cm^{-1}): 3393, 1585, 1488, 1316. **HRMS** (ESI): calc for $C_{13}H_{12}N^{35}Cl$ *m/z*: 217.0658 found $[M+H]^+$ 218.0736.

***N*-(4-Chlorophenyl)-3-fluorobenzenamine (6b)**



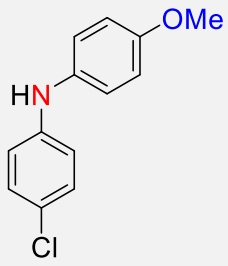
The general experimental procedure described for the synthesis of **6a-6e** was followed using 4-chloroaniline **4** (0.20 g, 1.5677 mmol, 1.0 eq.), 3-fluorophenylboronic acid **5b** (0.33 g, 2.3516 mmol, 1.5 eq.), pyridine (0.12 g, 1.5677 mmol, 1.0 eq.), **Moiras complex A** (17.1 mg, 0.0392 mmol, 0.025 eq.) and PhMe (5 mL). Compound **6b** was obtained as a brown oil (0.31 g, 1.4109 mmol, 90%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.24 (d, *J* = 8.9 Hz, 2H), 7.16–7.22 (m, 1H), 7.02 (d, *J* = 8.9 Hz, 2H), 6.70–6.77 (m, 2H), 6.57–6.64 (m, 1H), 5.72 (s, 1H, *N*-H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 163.9 (d, J_{C-F} = 244.5 Hz, 1C), 145.0 (d, J_{C-F} = 10.4 Hz, 1C), 140.8, 130.7 (d, J_{C-F} = 9.9 Hz, 1C), 129.5, 126.9, 120.2, 112.9 (d, J_{C-F} = 2.6 Hz, 1C), 107.7 (d, J_{C-F} = 21.6 Hz, 1C), 104.0 (d, J_{C-F} = 25.0 Hz, 1C). **^{19}F NMR** (377 MHz, $CDCl_3$) δ -111.9. **IR** (neat) V_{max} (cm^{-1}): 3410, 1585, 1484, 1323. **HRMS** (ESI): calc for $C_{12}H_9NF^{35}Cl$ *m/z*: 221.0408 found $[M+H]^+$ 222.0486.

N-(4-Chlorophenyl)-4-fluorobenzenamine (6c)



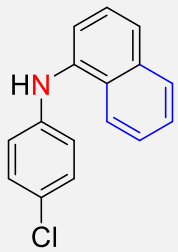
The general experimental procedure described for the synthesis of **6a-6e** was followed using 4-chloroaniline **4** (0.20 g, 1.5677 mmol, 1.0 eq.), 4-fluorophenylboronic acid **5c** (0.33 g, 2.3516 mmol, 1.5 eq.), pyridine (0.12 g, 1.5677 mmol, 1.0 eq.), **Moiras complex A** (17.1 mg, 0.0392 mmol, 0.025 eq.) and PhMe (5 mL). Compound **6c** was obtained as a brown oil (0.30 g, 1.3639 mmol, 87%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.19 (d, *J* = 8.8 Hz, 2H), 6.96–7.06 (m, 4H), 6.89 (d, *J* = 8.8 Hz, 2H), 5.54 (s, 1H, *N*-H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.4 (d, $J_{C\text{-}F}$ = 241.0 Hz, 1C), 142.8, 138.6 (d, $J_{C\text{-}F}$ = 2.6 Hz, 1C), 129.4, 125.2, 121.1 (d, $J_{C\text{-}F}$ = 7.9 Hz, 1C), 117.9, 116.2 (d, $J_{C\text{-}F}$ = 22.6 Hz, 1C). **^{19}F NMR** (377 MHz, $CDCl_3$) δ -121.1. **IR** (neat) V_{max} (cm^{-1}): 3409, 1593, 1489, 1312. **HRMS** (ESI): calc for $C_{12}H_9NF^{35}Cl$ *m/z*: 221.0408 found $[M+H]^+$ 222.0486.

4-Chloro-*N*-(4-methoxyphenyl)benzenamine (6d)



The general experimental procedure described for the synthesis of **6a-6e** was followed using 4-chloroaniline **4** (0.20 g, 1.5677 mmol, 1.0 eq.), 4-methoxyphenylboronic acid **5d** (0.36 g, 2.3516 mmol, 1.5 eq.), pyridine (0.12 g, 1.5677 mmol, 1.0 eq.), **Moiras complex A** (17.1 mg, 0.0392 mmol, 0.025 eq.) and PhMe (5 mL). Compound **6d** was obtained as a brown solid (0.31 g, 1.3325 mmol, 85%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.14 (d, *J* = 8.9 Hz, 2H), 7.05 (d, *J* = 8.9 Hz, 2H), 6.87 (d, *J* = 8.9 Hz, 2H), 6.82 (d, *J* = 8.9 Hz, 2H), 5.46 (s, 1H, *N*-H), 3.80 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 129.2, 122.7, 119.6, 116.7, 114.9, 55.7. **IR** (neat) V_{max} (cm^{-1}): 3415, 1595, 1491, 1292. **HRMS** (ESI): calc for $C_{13}H_{12}NO^{35}Cl$ *m/z*: 233.0607 found $[M+H]^+$ 234.0685. **MP**: 50-52°C

_N_-(4-Chlorophenyl)-1-naphthalenamine (6e)



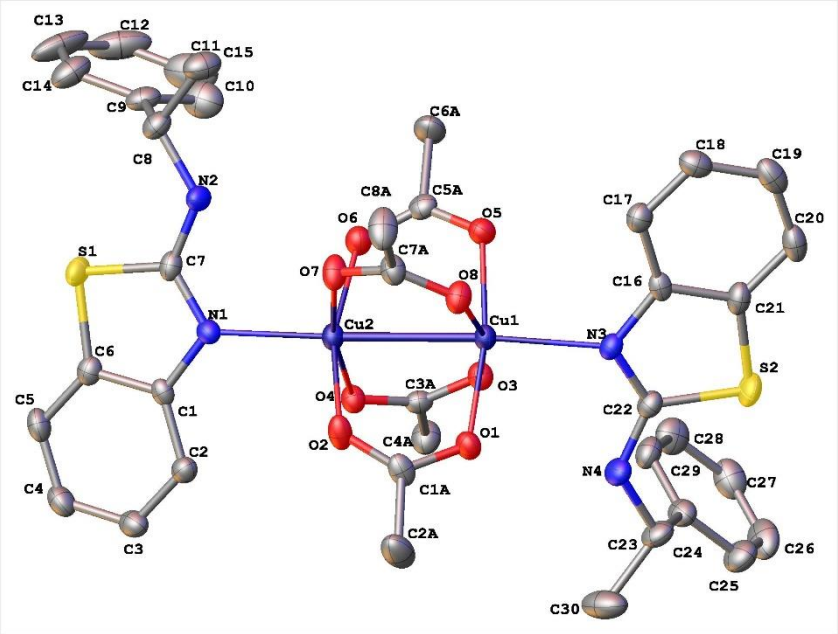
The general experimental procedure described for the synthesis of **6a**-**6e** was followed using 4-chloroaniline **4** (0.20 g, 1.5677 mmol, 1.0 eq.), 4-methoxyphenylboronic acid **5e** (0.36 g, 2.3516 mmol, 1.5 eq.), pyridine (0.12 g, 1.5677 mmol, 1.0 eq.), **Moiras complex A** (17.1 mg, 0.0392 mmol, 0.025 eq.) and PhMe (5 mL). Compound **6e** was obtained as a brown solid (0.35 g, 1.3953 mmol, 89%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.72–7.78 (m, 2H), 7.62–7.68 (m, 1H), 7.37–7.46 (m, 2H), 7.30–7.34 (m, 1H), 7.23–7.27 (m, 2H), 7.18–7.20 (m, 1H), 7.05–7.09 (m, 2H), 5.82 (s, 1H, *N*-H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.6, 140.3, 134.6, 129.6, 129.54, 129.52, 127.8, 126.74, 126.72, 126.3, 124.0, 120.1, 119.5, 112.5. **IR** (neat) V_{max} (cm^{-1}): 3396, 1591, 1487, 1311. **HRMS** (ESI): calc for $C_{16}H_{12}N^{35}Cl$ *m/z*: 253.0658 found $[M+H]^+$ 254.0736. **MP**: 90-92˚C

References

1. (a) S. Racioppi, L. Orian, C. Tubaro, A. Gennaro, and A. Ahmed Isse, Solvent Coordination Effect on Copper-Based Molecular Catalysts for Controlled Radical Polymerization, *Catalysts* 2022, **12**, 1656. (b) H. Zhang and R. M. Gschwind, Influence of Copper Salts, Solvents, and Ligands on the Structures of Precatalytic Phosphoramidite Copper Complexes for Conjugate Addition Reactions, *Chem. Eur. J.* 2007, **13**, 6691–6700. (c) H.-C Liang, M. J. Henson, L. Q. Hatcher, M. A. Vance, C. X. Zhang, D. Lahti, S. Kaderli, R. D. Sommer, A. L. Rheingold, A. D. ZuberbUhler, E. I. Solomon, and K. D. Karlin, *Inorg. Chem*. 2004, **43**, 4115−4117.

X-ray Crystallography

Structure report for Moiras Complex A (2023GU0007 /Expt 50 +Cu(OAc)2)



View showing the structure and atom labelling scheme. Atomic displacement ellipsoids drawn at 50% probability level, H- atoms omitted for clarity.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Results and discussion

Computing details

Data collection: *APEX3* Ver. 2016.9-0 (Bruker-AXS, 2016); cell refinement: *SAINT* V8.40B (?, 2016); data reduction: *SAINT* V8.40B (?, 2016); program(s) used to solve structure: SHELXT 2018/2 (Sheldrick, 2018); program(s) used to refine structure: *SHELXL* 2018/3 (Sheldrick, 2015); molecular graphics: Olex2 1.5 (Dolomanov *et al.*, 2009); software used to prepare material for publication: Olex2 1.5 (Dolomanov *et al.*, 2009).

References

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Sheldrick, G. M. (2015). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

(mo_2023gu0007_150k_0m)

Crystal data

$C_{38}H_{40}Cu_2N_4O_8S_2$	D_x = 1.467 Mg m^{-3}
M_r = 871.94	Mo $K\alpha$ radiation, λ = 0.71073 Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 9883 reflections
a = 10.1971 (4) Å	θ = 2.5–27.5°
b = 14.3001 (5) Å	μ = 1.24 mm^{-1}
c = 27.0785 (11) Å	T = 150 K
V = 3948.6 (3) Å^3	Block, green
Z = 4	0.25 × 0.14 × 0.05 mm
F(000) = 1800	

Data collection

Bruker D8 VENTURE diffractometer	9774 independent reflections
Radiation source: microfocus sealed tube, INCOATEC Iμs 3.0	8253 reflections with $I > 2\sigma(I)$
Multilayer mirror optics monochromator	R_{int} = 0.055
Detector resolution: 7.4074 pixels mm^{-1}	θ_{max} = 28.3°, θ_{min} = 2.1°
ϕ and ω scans	h = -13→9
Absorption correction: multi-scan *SADABS2016*/2 (Bruker,2016/2) was used for absorption correction. wR2(int) was 0.1188 before and 0.0705 after correction. The Ratio of minimum to maximum transmission is 0.8168. The λ/2 correction factor is Not present.	k = -17→19
T_{min} = 0.609, T_{max} = 0.746	l = -36→29
31215 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.041	$w = 1/[\sigma^2(F_o^2) + (0.0378P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.090	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.01	$\Delta\rangle_{max}$ = 0.59 e Å^{-3}
9774 reflections	$\Delta\rangle_{min}$ = -0.28 e Å^{-3}
494 parameters	Absolute structure: Refined as an inversion twin.
0 restraints	Absolute structure parameter: 0.019 (13)
Primary atom site location: dual	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.
Refinement. Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for (mo_2023gu0007_150k_0m)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.25470 (5)	0.34441 (3)	0.39415 (2)	0.01857 (11)
S1	0.46804 (11)	0.82573 (7)	0.42404 (4)	0.0254 (2)
O1	0.1043 (3)	0.35952 (19)	0.43933 (10)	0.0251 (7)
N1	0.3465 (3)	0.6663 (2)	0.43371 (11)	0.0197 (7)
C1	0.2540 (5)	0.7364 (2)	0.44131 (13)	0.0185 (8)
C1A	0.0979 (5)	0.4274 (3)	0.46952 (15)	0.0243 (10)
Cu2	0.30203 (5)	0.52111 (3)	0.41716 (2)	0.01903 (12)
S2	0.06214 (13)	0.07240 (8)	0.32956 (4)	0.0319 (3)
O2	0.1769 (3)	0.49394 (18)	0.47170 (10)	0.0280 (7)
N2	0.5715 (3)	0.6531 (2)	0.41884 (13)	0.0244 (7)
H2	0.565063	0.593182	0.419735	0.029*
C2	0.1218 (4)	0.7220 (3)	0.45217 (15)	0.0234 (9)
H2A	0.089104	0.661670	0.455852	0.028*
C2A	-0.0162 (6)	0.4264 (3)	0.50451 (19)	0.0425 (14)
H2AA	-0.093141	0.448838	0.487855	0.064*
H2AB	0.002548	0.465934	0.532259	0.064*
H2AC	-0.031147	0.363613	0.515801	0.064*
O3	0.1442 (3)	0.40517 (18)	0.34206 (9)	0.0220 (6)
N3	0.1987 (4)	0.2023 (2)	0.37296 (11)	0.0198 (7)
C3	0.0400 (5)	0.7985 (3)	0.45740 (15)	0.0278 (10)
H3	-0.048151	0.789433	0.464753	0.033*
C3A	0.1108 (4)	0.4904 (3)	0.34386 (14)	0.0208 (9)
O4	0.1526 (3)	0.54899 (18)	0.37427 (10)	0.0221 (6)
N4	-0.0265 (4)	0.2324 (2)	0.36971 (13)	0.0272 (8)
H4	-0.015293	0.281074	0.387880	0.033*
C4	0.0885 (5)	0.8895 (3)	0.45174 (16)	0.0299 (11)
H4A	0.031716	0.940087	0.454784	0.036*
C4A	0.0149 (5)	0.5236 (3)	0.30523 (16)	0.0314 (10)
H4AA	0.057864	0.526308	0.273716	0.047*

H4AB	-0.016514	0.584727	0.313908	0.047*
H4AC	-0.057615	0.480947	0.303494	0.047*
O5	0.4063 (3)	0.35455 (19)	0.35019 (10)	0.0245 (7)
C5	0.2182 (5)	0.9050 (3)	0.44180 (15)	0.0280 (11)
H5	0.250387	0.965496	0.438442	0.034*
C5A	0.4506 (4)	0.4335 (3)	0.33870 (15)	0.0227 (9)
O6	0.4180 (3)	0.51001 (19)	0.35796 (10)	0.0262 (7)
C6	0.3010 (5)	0.8284 (3)	0.43682 (14)	0.0222 (9)
C6A	0.5514 (5)	0.4365 (3)	0.29776 (17)	0.0352 (12)
H6AA	0.521004	0.476823	0.271827	0.053*
H6AB	0.564442	0.374631	0.284927	0.053*
H6AC	0.632838	0.459990	0.310544	0.053*
O7	0.4472 (3)	0.46832 (19)	0.45588 (10)	0.0273 (7)
C7	0.4626 (4)	0.7026 (3)	0.42552 (14)	0.0207 (9)
C7A	0.4509 (5)	0.3827 (3)	0.46700 (15)	0.0247 (10)
O8	0.3766 (3)	0.32018 (19)	0.45016 (10)	0.0257 (7)
C8	0.7009 (5)	0.6931 (3)	0.41010 (15)	0.0262 (9)
H8	0.719104	0.738216	0.436456	0.031*
C8A	0.5554 (6)	0.3534 (3)	0.50347 (17)	0.0382 (12)
H8AA	0.629592	0.394449	0.500599	0.057*
H8AB	0.582238	0.290432	0.496566	0.057*
H8AC	0.520867	0.356871	0.536397	0.057*
C9	0.7094 (5)	0.7439 (3)	0.36102 (15)	0.0288 (10)
C10	0.6618 (6)	0.7028 (4)	0.31854 (17)	0.0442 (14)
H10	0.620691	0.644849	0.320136	0.053*
C11	0.6752 (6)	0.7481 (5)	0.27322 (19)	0.0582 (18)
H11	0.644336	0.719721	0.244584	0.070*
C12	0.7337 (6)	0.8341 (5)	0.2707 (2)	0.0620 (19)
H12	0.743555	0.863767	0.240370	0.074*
C13	0.7774 (5)	0.8763 (5)	0.3128 (2)	0.0588 (18)
H13	0.814616	0.935604	0.311032	0.071*
C14	0.7670 (5)	0.8318 (3)	0.35822 (19)	0.0406 (12)
H14	0.798442	0.860636	0.386618	0.049*
C15	0.8010 (5)	0.6145 (3)	0.41381 (18)	0.0361 (11)
H15A	0.802840	0.591021	0.447015	0.054*
H15B	0.886152	0.637887	0.405125	0.054*
H15C	0.777086	0.564978	0.391622	0.054*
C16	0.2860 (5)	0.1319 (2)	0.36048 (13)	0.0200 (9)
C17	0.4196 (5)	0.1302 (3)	0.37107 (15)	0.0252 (10)
H17	0.458714	0.179823	0.387662	0.030*

C18	0.4931 (5)	0.0543 (3)	0.35673 (16)	0.0300 (11)
H18	0.582070	0.052658	0.364157	0.036*
C19	0.4370 (5)	-0.0195 (3)	0.33146 (16)	0.0335 (11)
H19	0.489066	-0.069719	0.321830	0.040*
C20	0.3054 (5)	-0.0200 (3)	0.32032 (15)	0.0318 (11)
H20	0.267946	-0.069433	0.303005	0.038*
C21	0.2304 (5)	0.0551 (3)	0.33561 (14)	0.0264 (10)
C22	0.0792 (5)	0.1798 (3)	0.36015 (14)	0.0219 (9)
C23	-0.1582 (5)	0.2142 (3)	0.35203 (16)	0.0307 (10)
H23	-0.181295	0.150041	0.361240	0.037*
C24	-0.1714 (5)	0.2227 (3)	0.29628 (16)	0.0271 (10)
C25	-0.2775 (5)	0.1801 (4)	0.27276 (18)	0.0380 (12)
H25	-0.338149	0.146451	0.291238	0.046*
C26	-0.2930 (6)	0.1875 (4)	0.22232 (19)	0.0440 (13)
H26	-0.364363	0.159189	0.207026	0.053*
C27	-0.2037 (6)	0.2366 (3)	0.19455 (18)	0.0385 (12)
H27	-0.213706	0.240807	0.160488	0.046*
C28	-0.0992 (5)	0.2794 (3)	0.21745 (18)	0.0369 (12)
H28	-0.039258	0.313268	0.198733	0.044*
C29	-0.0824 (5)	0.2726 (3)	0.26771 (16)	0.0292 (10)
H29	-0.010997	0.301516	0.282672	0.035*
C30	-0.2507 (6)	0.2800 (4)	0.37935 (19)	0.0507 (14)
H30A	-0.240556	0.271492	0.414310	0.076*
H30B	-0.339628	0.266453	0.370121	0.076*
H30C	-0.230336	0.343540	0.370846	0.076*

Atomic displacement parameters (Å^2) for (mo_2023gu0007_150k_0m)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0212 (3)	0.0171 (2)	0.0174 (2)	-0.0010 (2)	-0.0008 (2)	-0.00138 (17)
S1	0.0253 (6)	0.0171 (5)	0.0340 (6)	-0.0040 (4)	-0.0017 (5)	0.0027 (4)
O1	0.0293 (18)	0.0234 (15)	0.0225 (15)	-0.0008 (14)	0.0034 (13)	-0.0042 (12)
N1	0.0186 (18)	0.0205 (17)	0.0199 (17)	0.0014 (15)	-0.0023 (13)	-0.0026 (13)
C1	0.025 (2)	0.0176 (17)	0.0128 (17)	0.0025 (19)	-0.0010 (17)	-0.0012 (13)
C1A	0.037 (3)	0.020 (2)	0.015 (2)	0.004 (2)	0.0010 (18)	0.0032 (15)
Cu2	0.0230 (3)	0.0160 (2)	0.0181 (2)	-0.0009 (2)	-0.0026 (2)	-0.00011 (18)
S2	0.0324 (7)	0.0271 (5)	0.0362 (7)	-0.0079 (5)	-0.0068 (5)	-0.0092 (5)
O2	0.045 (2)	0.0193 (14)	0.0194 (14)	-0.0036 (14)	0.0046 (13)	-0.0013 (11)
N2	0.022 (2)	0.0177 (15)	0.0336 (19)	-0.0009 (15)	0.0032 (16)	0.0013 (15)

C2	0.024 (2)	0.025 (2)	0.022 (2)	-0.0022 (19)	0.0012 (18)	-0.0023 (16)
C2A	0.052 (4)	0.033 (2)	0.042 (3)	0.000 (3)	0.022 (3)	-0.001 (2)
O3	0.0255 (17)	0.0211 (14)	0.0195 (15)	0.0009 (13)	-0.0023 (12)	-0.0004 (11)
N3	0.0207 (18)	0.0205 (16)	0.0181 (16)	-0.0038 (16)	-0.0030 (15)	-0.0004 (12)
C3	0.023 (2)	0.034 (2)	0.027 (2)	0.002 (2)	-0.0018 (19)	-0.0071 (18)
C3A	0.018 (2)	0.025 (2)	0.020 (2)	0.0003 (18)	0.0009 (17)	0.0027 (16)
O4	0.0250 (17)	0.0200 (14)	0.0212 (14)	0.0011 (13)	-0.0028 (12)	-0.0016 (11)
N4	0.022 (2)	0.0312 (19)	0.0282 (19)	-0.0031 (17)	-0.0039 (16)	-0.0083 (15)
C4	0.032 (3)	0.027 (2)	0.031 (2)	0.008 (2)	-0.003 (2)	-0.0061 (18)
C4A	0.032 (3)	0.029 (2)	0.033 (2)	0.000 (2)	-0.010 (2)	-0.0004 (19)
O5	0.0249 (18)	0.0234 (14)	0.0253 (15)	0.0006 (14)	0.0028 (13)	-0.0008 (12)
C5	0.037 (3)	0.0184 (19)	0.028 (2)	0.003 (2)	-0.004 (2)	-0.0037 (15)
C5A	0.019 (2)	0.030 (2)	0.020 (2)	-0.0026 (19)	-0.0040 (17)	-0.0002 (17)
O6	0.0291 (19)	0.0240 (15)	0.0255 (16)	-0.0022 (14)	0.0016 (13)	-0.0020 (12)
C6	0.026 (2)	0.022 (2)	0.0180 (18)	-0.0031 (19)	-0.0023 (17)	0.0005 (15)
C6A	0.036 (3)	0.036 (2)	0.033 (3)	-0.004 (2)	0.015 (2)	-0.0060 (19)
O7	0.0359 (19)	0.0183 (14)	0.0278 (16)	-0.0002 (14)	-0.0117 (14)	0.0009 (12)
C7	0.026 (2)	0.0173 (18)	0.019 (2)	-0.0029 (18)	-0.0056 (17)	0.0016 (15)
C7A	0.030 (3)	0.023 (2)	0.021 (2)	0.002 (2)	-0.0050 (19)	-0.0017 (16)
O8	0.0312 (19)	0.0213 (15)	0.0247 (15)	-0.0021 (14)	-0.0071 (13)	0.0022 (11)
C8	0.023 (2)	0.030 (2)	0.026 (2)	-0.006 (2)	-0.0025 (19)	0.0001 (17)
C8A	0.047 (3)	0.027 (2)	0.041 (3)	0.004 (2)	-0.023 (2)	0.002 (2)
C9	0.017 (2)	0.041 (2)	0.028 (2)	-0.001 (2)	0.0002 (19)	0.0040 (18)
C10	0.042 (3)	0.059 (3)	0.032 (3)	0.005 (3)	-0.008 (2)	-0.002 (2)
C11	0.047 (4)	0.102 (5)	0.025 (3)	0.013 (4)	-0.007 (3)	-0.001 (3)
C12	0.028 (3)	0.112 (5)	0.046 (3)	0.008 (4)	0.006 (3)	0.040 (3)
C13	0.023 (3)	0.081 (4)	0.072 (4)	-0.010 (3)	0.002 (3)	0.047 (3)
C14	0.027 (3)	0.049 (3)	0.047 (3)	-0.010 (3)	-0.005 (2)	0.013 (2)
C15	0.024 (2)	0.043 (3)	0.041 (3)	0.000 (2)	0.000 (2)	0.009 (2)
C16	0.030 (3)	0.0165 (17)	0.0136 (18)	0.0004 (17)	0.0003 (17)	0.0012 (13)
C17	0.028 (3)	0.023 (2)	0.024 (2)	-0.0026 (19)	-0.0015 (19)	0.0010 (16)
C18	0.028 (3)	0.033 (2)	0.029 (2)	0.007 (2)	0.003 (2)	0.0023 (19)
C19	0.049 (3)	0.026 (2)	0.026 (2)	0.009 (2)	0.007 (2)	0.0015 (19)
C20	0.049 (3)	0.021 (2)	0.025 (2)	-0.001 (2)	0.001 (2)	-0.0035 (17)
C21	0.034 (3)	0.0220 (19)	0.023 (2)	-0.0058 (19)	-0.0028 (19)	-0.0019 (16)
C22	0.022 (2)	0.024 (2)	0.020 (2)	-0.0038 (18)	-0.0038 (17)	-0.0007 (16)
C23	0.019 (2)	0.040 (2)	0.033 (2)	-0.006 (2)	-0.0031 (19)	-0.002 (2)
C24	0.022 (3)	0.031 (2)	0.027 (2)	0.001 (2)	-0.0044 (18)	-0.0052 (17)
C25	0.028 (3)	0.045 (3)	0.041 (3)	-0.007 (2)	-0.006 (2)	-0.005 (2)
C26	0.041 (3)	0.044 (3)	0.047 (3)	0.004 (3)	-0.023 (3)	-0.015 (2)

C27	0.050 (3)	0.036 (3)	0.029 (2)	0.009 (3)	-0.012 (2)	-0.002 (2)
C28	0.043 (3)	0.031 (2)	0.037 (3)	0.002 (2)	-0.004 (2)	0.008 (2)
C29	0.028 (3)	0.024 (2)	0.035 (3)	-0.005 (2)	-0.007 (2)	-0.0005 (18)
C30	0.027 (3)	0.085 (4)	0.041 (3)	0.002 (3)	0.002 (2)	-0.015 (3)

Geometric parameters (Å, º) for (mo_2023gu0007_150k_0m)

Cu1—O1	1.974 (3)	C6A—H6AC	0.9600
Cu1—Cu2	2.6470 (6)	O7—C7A	1.261 (5)
Cu1—O3	2.004 (3)	C7A—O8	1.258 (5)
Cu1—N3	2.188 (3)	C7A—C8A	1.513 (6)
Cu1—O5	1.956 (3)	C8—H8	0.9800
Cu1—O8	1.991 (3)	C8—C9	1.517 (6)
S1—C6	1.739 (5)	C8—C15	1.521 (6)
S1—C7	1.761 (4)	C8A—H8AA	0.9600
O1—C1A	1.271 (5)	C8A—H8AB	0.9600
N1—C1	1.392 (5)	C8A—H8AC	0.9600
N1—Cu2	2.172 (3)	C9—C10	1.380 (6)
N1—C7	1.312 (5)	C9—C14	1.389 (7)
C1—C2	1.395 (6)	C10—H10	0.9300
C1—C6	1.405 (5)	C10—C11	1.394 (7)
C1A—O2	1.248 (5)	C11—H11	0.9300
C1A—C2A	1.501 (6)	C11—C12	1.369 (9)
Cu2—O2	1.990 (3)	C12—H12	0.9300
Cu2—O4	1.957 (3)	C12—C13	1.364 (9)
Cu2—O6	1.998 (3)	C13—H13	0.9300
Cu2—O7	1.965 (3)	C13—C14	1.390 (7)
S2—C21	1.741 (5)	C14—H14	0.9300
S2—C22	1.754 (4)	C15—H15A	0.9600
N2—H2	0.8600	C15—H15B	0.9600
N2—C7	1.329 (5)	C15—H15C	0.9600
N2—C8	1.458 (6)	C16—C17	1.393 (7)
C2—H2A	0.9300	C16—C21	1.408 (5)
C2—C3	1.383 (6)	C17—H17	0.9300
C2A—H2AA	0.9600	C17—C18	1.374 (6)
C2A—H2AB	0.9600	C18—H18	0.9300
C2A—H2AC	0.9600	C18—C19	1.382 (6)
O3—C3A	1.266 (5)	C19—H19	0.9300
N3—C16	1.385 (5)	C19—C20	1.376 (7)
N3—C22	1.306 (6)	C20—H20	0.9300

C3—H3	0.9300	C20—C21	1.382 (6)
C3—C4	1.400 (6)	C23—H23	0.9800
C3A—O4	1.250 (5)	C23—C24	1.521 (6)
C3A—C4A	1.509 (6)	C23—C30	1.524 (7)
N4—H4	0.8600	C24—C25	1.395 (6)
N4—C22	1.340 (6)	C24—C29	1.389 (6)
N4—C23	1.449 (6)	C25—H25	0.9300
C4—H4A	0.9300	C25—C26	1.379 (7)
C4—C5	1.368 (7)	C26—H26	0.9300
C4A—H4AA	0.9600	C26—C27	1.374 (8)
C4A—H4AB	0.9600	C27—H27	0.9300
C4A—H4AC	0.9600	C27—C28	1.377 (7)
O5—C5A	1.256 (5)	C28—H28	0.9300
C5—H5	0.9300	C28—C29	1.375 (6)
C5—C6	1.389 (6)	C29—H29	0.9300
C5A—O6	1.256 (5)	C30—H30A	0.9600
C5A—C6A	1.513 (6)	C30—H30B	0.9600
C6A—H6AA	0.9600	C30—H30C	0.9600
C6A—H6AB	0.9600		
O1—Cu1—Cu2	83.76 (8)	C7A—O7—Cu2	121.5 (3)
O1—Cu1—O3	87.23 (12)	N1—C7—S1	115.3 (3)
O1—Cu1—N3	93.51 (13)	N1—C7—N2	124.5 (3)
O1—Cu1—O8	91.84 (12)	N2—C7—S1	120.2 (3)
O3—Cu1—Cu2	81.62 (8)	O7—C7A—C8A	116.5 (4)
O3—Cu1—N3	94.09 (12)	O8—C7A—O7	125.9 (4)
N3—Cu1—Cu2	175.00 (10)	O8—C7A—C8A	117.7 (4)
O5—Cu1—O1	169.42 (12)	C7A—O8—Cu1	121.9 (3)
O5—Cu1—Cu2	85.89 (8)	N2—C8—H8	108.2
O5—Cu1—O3	89.07 (12)	N2—C8—C9	112.4 (4)
O5—Cu1—N3	96.64 (13)	N2—C8—C15	107.9 (4)
O5—Cu1—O8	89.04 (13)	C9—C8—H8	108.1
O8—Cu1—Cu2	82.71 (8)	C9—C8—C15	111.9 (4)
O8—Cu1—O3	164.30 (11)	C15—C8—H8	108.2
O8—Cu1—N3	101.61 (12)	C7A—C8A—H8AA	109.5
C6—S1—C7	89.2 (2)	C7A—C8A—H8AB	109.5
C1A—O1—Cu1	121.5 (3)	C7A—C8A—H8AC	109.5
C1—N1—Cu2	125.3 (3)	H8AA—C8A—H8AB	109.5
C7—N1—C1	110.6 (3)	H8AA—C8A—H8AC	109.5
C7—N1—Cu2	122.1 (3)	H8AB—C8A—H8AC	109.5

N1—C1—C2	125.4 (4)	C10—C9—C8	120.4 (4)
N1—C1—C6	115.5 (4)	C10—C9—C14	119.2 (4)
C2—C1—C6	119.1 (4)	C14—C9—C8	120.3 (4)
O1—C1A—C2A	116.0 (4)	C9—C10—H10	119.9
O2—C1A—O1	125.4 (4)	C9—C10—C11	120.1 (6)
O2—C1A—C2A	118.6 (4)	C11—C10—H10	119.9
N1—Cu2—Cu1	177.76 (9)	C10—C11—H11	119.9
O2—Cu2—Cu1	82.61 (8)	C12—C11—C10	120.2 (5)
O2—Cu2—N1	99.63 (12)	C12—C11—H11	119.9
O2—Cu2—O6	163.58 (11)	C11—C12—H12	120.0
O4—Cu2—Cu1	85.00 (8)	C13—C12—C11	119.9 (5)
O4—Cu2—N1	95.17 (12)	C13—C12—H12	120.0
O4—Cu2—O2	88.91 (13)	C12—C13—H13	119.6
O4—Cu2—O6	90.03 (13)	C12—C13—C14	120.8 (6)
O4—Cu2—O7	168.99 (11)	C14—C13—H13	119.6
O6—Cu2—Cu1	80.98 (8)	C9—C14—C13	119.7 (5)
O6—Cu2—N1	96.78 (12)	C9—C14—H14	120.2
O7—Cu2—Cu1	84.04 (8)	C13—C14—H14	120.2
O7—Cu2—N1	95.74 (12)	C8—C15—H15A	109.5
O7—Cu2—O2	90.69 (13)	C8—C15—H15B	109.5
O7—Cu2—O6	87.24 (13)	C8—C15—H15C	109.5
C21—S2—C22	89.0 (2)	H15A—C15—H15B	109.5
C1A—O2—Cu2	121.8 (3)	H15A—C15—H15C	109.5
C7—N2—H2	117.6	H15B—C15—H15C	109.5
C7—N2—C8	124.7 (3)	N3—C16—C17	126.3 (4)
C8—N2—H2	117.6	N3—C16—C21	115.1 (4)
C1—C2—H2A	120.4	C17—C16—C21	118.6 (4)
C3—C2—C1	119.2 (4)	C16—C17—H17	120.3
C3—C2—H2A	120.4	C18—C17—C16	119.3 (4)
C1A—C2A—H2AA	109.5	C18—C17—H17	120.3
C1A—C2A—H2AB	109.5	C17—C18—H18	119.4
C1A—C2A—H2AC	109.5	C17—C18—C19	121.2 (5)
H2AA—C2A—H2AB	109.5	C19—C18—H18	119.4
H2AA—C2A—H2AC	109.5	C18—C19—H19	119.5
H2AB—C2A—H2AC	109.5	C20—C19—C18	121.1 (4)
C3A—O3—Cu1	122.8 (3)	C20—C19—H19	119.5
C16—N3—Cu1	124.8 (3)	C19—C20—H20	121.0
C22—N3—Cu1	122.8 (3)	C19—C20—C21	118.1 (4)
C22—N3—C16	110.8 (3)	C21—C20—H20	121.0
C2—C3—H3	119.6	C16—C21—S2	109.3 (3)

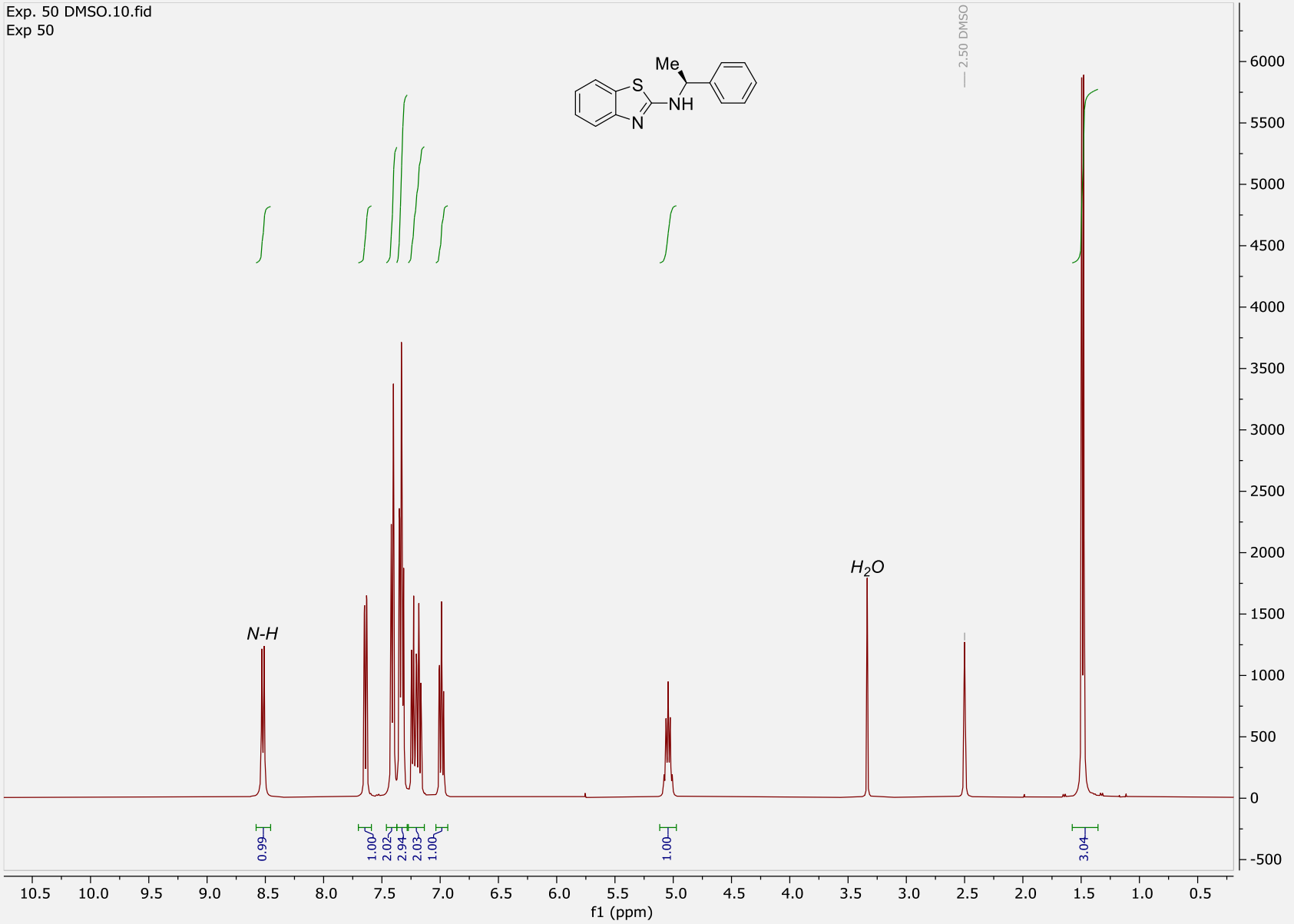
C2—C3—C4	120.8 (5)	C20—C21—S2	128.9 (3)
C4—C3—H3	119.6	C20—C21—C16	121.8 (4)
O3—C3A—C4A	116.8 (4)	N3—C22—S2	115.7 (3)
O4—C3A—O3	125.4 (4)	N3—C22—N4	124.1 (4)
O4—C3A—C4A	117.8 (4)	N4—C22—S2	120.2 (3)
C3A—O4—Cu2	121.3 (3)	N4—C23—H23	107.8
C22—N4—H4	117.2	N4—C23—C24	113.3 (4)
C22—N4—C23	125.5 (4)	N4—C23—C30	107.6 (4)
C23—N4—H4	117.2	C24—C23—H23	107.8
C3—C4—H4A	119.6	C24—C23—C30	112.2 (4)
C5—C4—C3	120.9 (4)	C30—C23—H23	107.8
C5—C4—H4A	119.6	C25—C24—C23	119.1 (4)
C3A—C4A—H4AA	109.5	C29—C24—C23	122.4 (4)
C3A—C4A—H4AB	109.5	C29—C24—C25	118.5 (4)
C3A—C4A—H4AC	109.5	C24—C25—H25	119.8
H4AA—C4A—H4AB	109.5	C26—C25—C24	120.5 (5)
H4AA—C4A—H4AC	109.5	C26—C25—H25	119.8
H4AB—C4A—H4AC	109.5	C25—C26—H26	119.8
C5A—O5—Cu1	120.1 (3)	C27—C26—C25	120.3 (5)
C4—C5—H5	120.7	C27—C26—H26	119.8
C4—C5—C6	118.6 (4)	C26—C27—H27	120.2
C6—C5—H5	120.7	C26—C27—C28	119.6 (5)
O5—C5A—O6	125.8 (4)	C28—C27—H27	120.2
O5—C5A—C6A	116.8 (4)	C27—C28—H28	119.6
O6—C5A—C6A	117.4 (4)	C29—C28—C27	120.7 (5)
C5A—O6—Cu2	124.0 (3)	C29—C28—H28	119.6
C1—C6—S1	109.3 (3)	C24—C29—H29	119.8
C5—C6—S1	129.2 (3)	C28—C29—C24	120.4 (4)
C5—C6—C1	121.5 (4)	C28—C29—H29	119.8
C5A—C6A—H6AA	109.5	C23—C30—H30A	109.5
C5A—C6A—H6AB	109.5	C23—C30—H30B	109.5
C5A—C6A—H6AC	109.5	C23—C30—H30C	109.5
H6AA—C6A—H6AB	109.5	H30A—C30—H30B	109.5
H6AA—C6A—H6AC	109.5	H30A—C30—H30C	109.5
H6AB—C6A—H6AC	109.5	H30B—C30—H30C	109.5
Cu1—O1—C1A—O2	5.7 (6)	C7—N1—C1—C2	-178.3 (4)
Cu1—O1—C1A—C2A	-175.2 (3)	C7—N1—C1—C6	2.4 (5)
Cu1—O3—C3A—O4	8.3 (6)	C7—N2—C8—C9	-66.3 (5)

Cu1—O3—C3A—C4A	-173.2 (3)	C7—N2—C8—C15	169.8 (4)
Cu1—N3—C16—C17	17.7 (5)	C8—N2—C7—S1	0.1 (5)
Cu1—N3—C16—C21	-163.6 (3)	C8—N2—C7—N1	-179.5 (4)
Cu1—N3—C22—S2	164.13 (18)	C8—C9—C10—C11	-177.1 (5)
Cu1—N3—C22—N4	-17.0 (6)	C8—C9—C14—C13	178.2 (5)
Cu1—O5—C5A—O6	10.1 (6)	C8A—C7A—O8—Cu1	-169.9 (3)
Cu1—O5—C5A—C6A	-169.4 (3)	C9—C10—C11—C12	-1.1 (9)
O1—C1A—O2—Cu2	15.5 (6)	C10—C9—C14—C13	-0.8 (8)
N1—C1—C2—C3	-178.1 (4)	C10—C11—C12—C13	-0.9 (9)
N1—C1—C6—S1	-1.4 (4)	C11—C12—C13—C14	2.0 (9)
N1—C1—C6—C5	177.8 (3)	C12—C13—C14—C9	-1.1 (8)
C1—N1—C7—S1	-2.3 (4)	C14—C9—C10—C11	1.9 (8)
C1—N1—C7—N2	177.4 (4)	C15—C8—C9—C10	74.6 (6)
C1—C2—C3—C4	0.2 (6)	C15—C8—C9—C14	-104.4 (5)
Cu2—N1—C1—C2	17.6 (5)	C16—N3—C22—S2	-2.2 (4)
Cu2—N1—C1—C6	-161.7 (3)	C16—N3—C22—N4	176.7 (4)
Cu2—N1—C7—S1	162.39 (18)	C16—C17—C18—C19	0.9 (6)
Cu2—N1—C7—N2	-17.9 (5)	C17—C16—C21—S2	177.3 (3)
Cu2—O7—C7A—O8	10.1 (7)	C17—C16—C21—C20	-1.9 (6)
Cu2—O7—C7A—C8A	-170.6 (3)	C17—C18—C19—C20	-0.8 (7)
N2—C8—C9—C10	-47.1 (6)	C18—C19—C20—C21	-0.7 (7)
N2—C8—C9—C14	134.0 (4)	C19—C20—C21—S2	-177.0 (3)
C2—C1—C6—S1	179.3 (3)	C19—C20—C21—C16	2.1 (6)
C2—C1—C6—C5	-1.5 (6)	C21—S2—C22—N3	1.2 (3)
C2—C3—C4—C5	-1.2 (7)	C21—S2—C22—N4	-177.8 (4)
C2A—C1A—O2—Cu2	-163.5 (3)	C21—C16—C17—C18	0.5 (6)
O3—C3A—O4—Cu2	11.0 (6)	C22—S2—C21—C16	0.2 (3)
N3—C16—C17—C18	179.1 (4)	C22—S2—C21—C20	179.3 (4)
N3—C16—C21—S2	-1.5 (4)	C22—N3—C16—C17	-176.3 (4)
N3—C16—C21—C20	179.3 (4)	C22—N3—C16—C21	2.4 (5)
C3—C4—C5—C6	0.8 (7)	C22—N4—C23—C24	-65.8 (6)
N4—C23—C24—C25	160.2 (4)	C22—N4—C23—C30	169.5 (4)
N4—C23—C24—C29	-20.7 (6)	C23—N4—C22—S2	-7.4 (6)

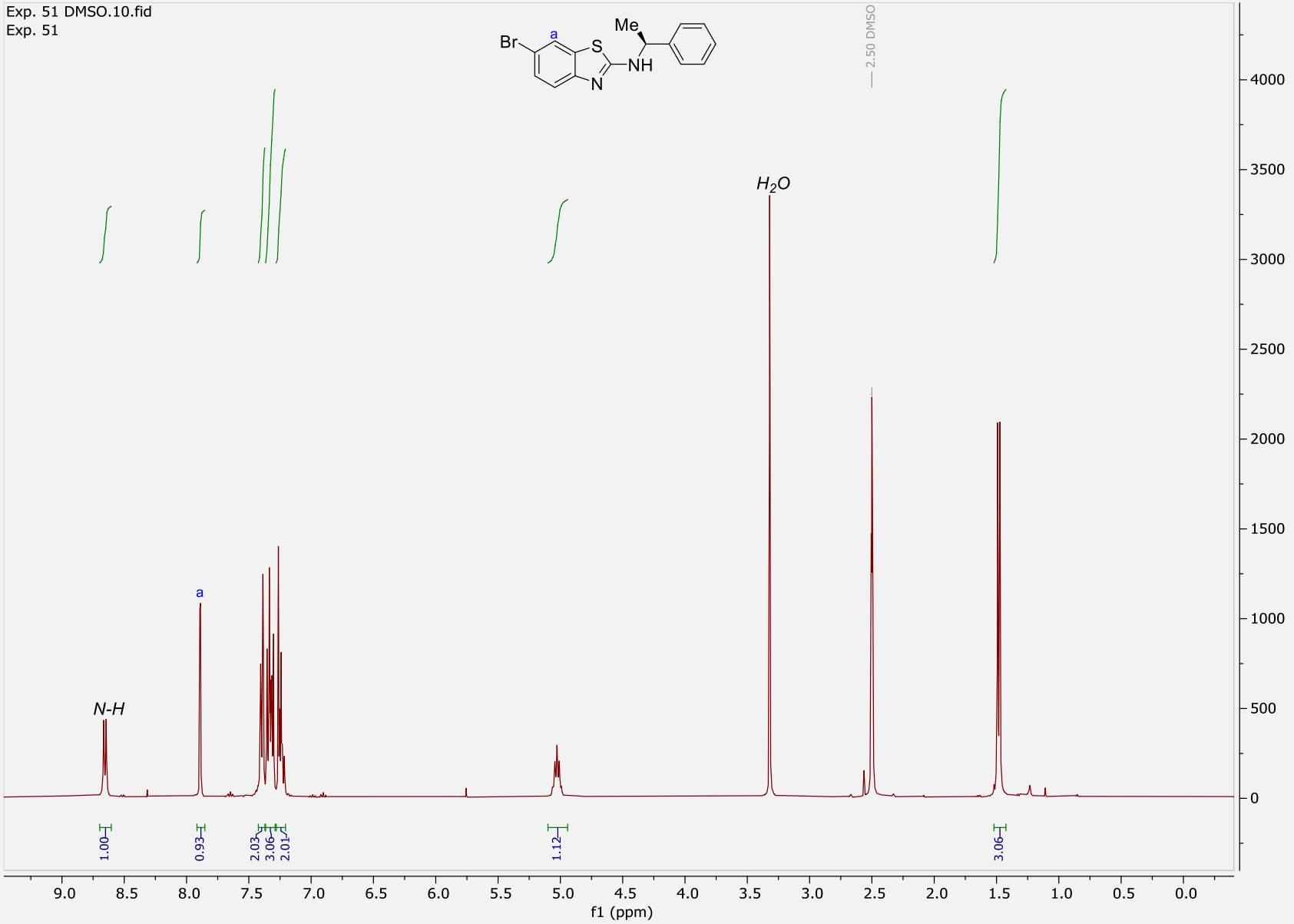
C4—C5—C6—S1	179.6 (3)	C23—N4—C22—N3	173.7 (4)
C4—C5—C6—C1	0.6 (6)	C23—C24—C25—C26	179.2 (4)
C4A—C3A—O4—Cu2	-167.5 (3)	C23—C24—C29—C28	-179.2 (4)
O5—C5A—O6—Cu2	7.5 (6)	C24—C25—C26—C27	0.4 (8)
C6—S1—C7—N1	1.3 (3)	C25—C24—C29—C28	0.0 (7)
C6—S1—C7—N2	-178.4 (3)	C25—C26—C27—C28	-0.8 (8)
C6—C1—C2—C3	1.1 (6)	C26—C27—C28—C29	0.8 (7)
C6A—C5A—O6—Cu2	-173.0 (3)	C27—C28—C29—C24	-0.4 (7)
O7—C7A—O8—Cu1	9.4 (7)	C29—C24—C25—C26	0.0 (7)
C7—S1—C6—C1	0.1 (3)	C30—C23—C24—C25	-77.7 (6)
C7—S1—C6—C5	-179.0 (4)	C30—C23—C24—C29	101.5 (5)

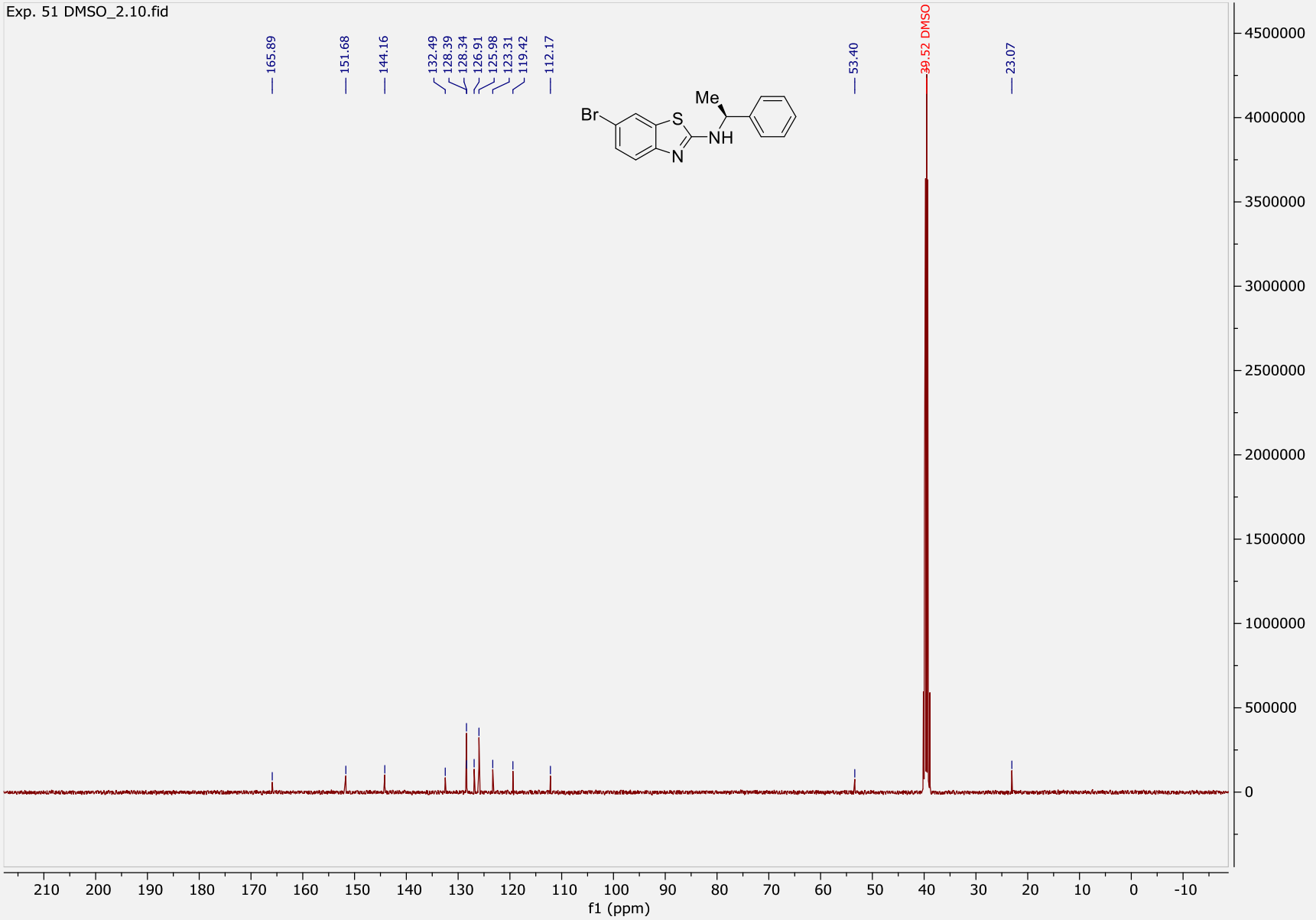
Document origin: *publCIF* [Westrip, S. P. (2010). *J. Apply. Cryst.*, **43**, 920-925].

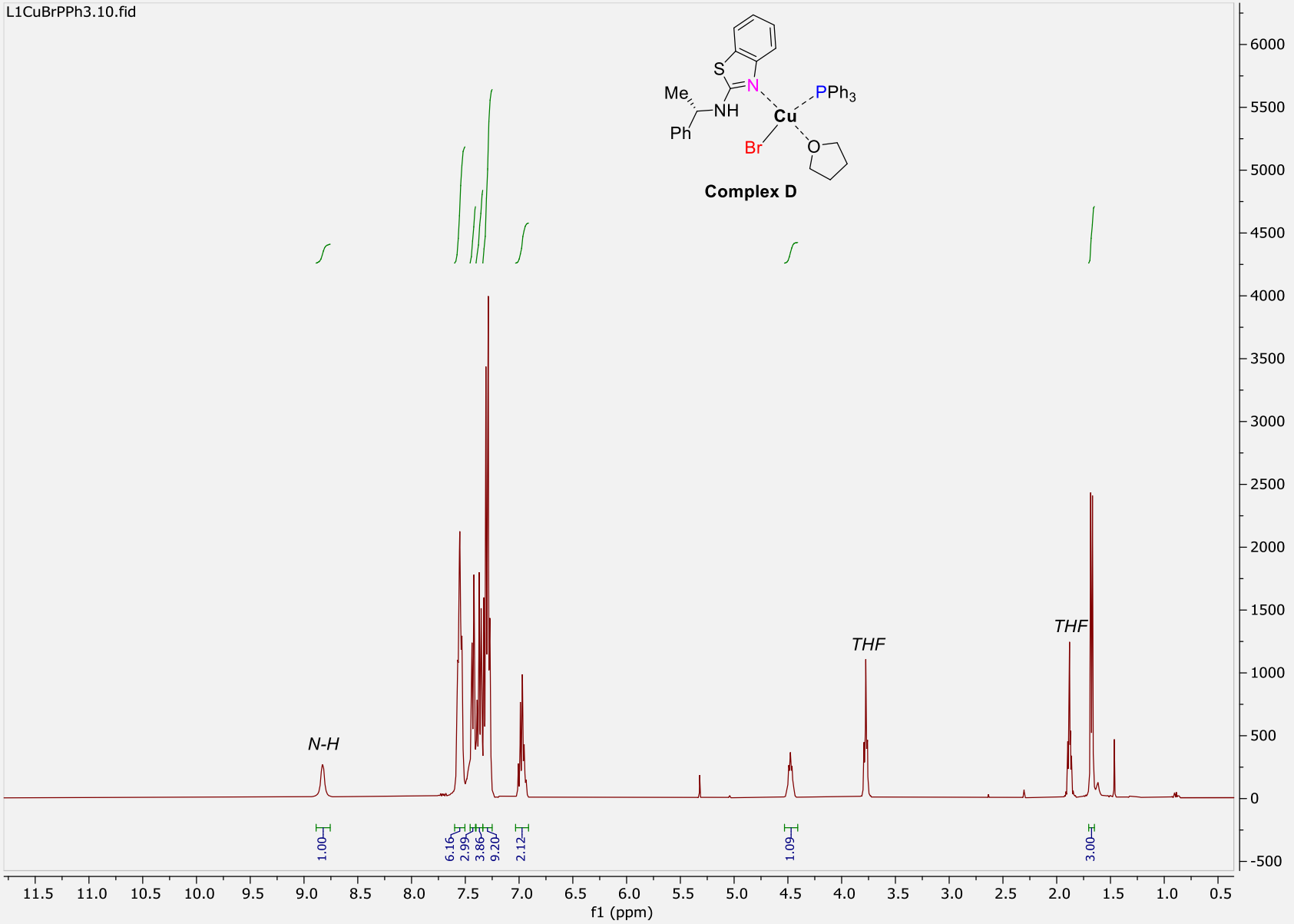
^{1}H and ^{13}C NMR Spectra


Exp. 50 DMSO.10.fid
Exp 50
Me
S
NH
N
2.50 DMSO
N-H
H2O
0.99
1.00
2.02
2.94
2.03
1.00
1.00
3.04
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

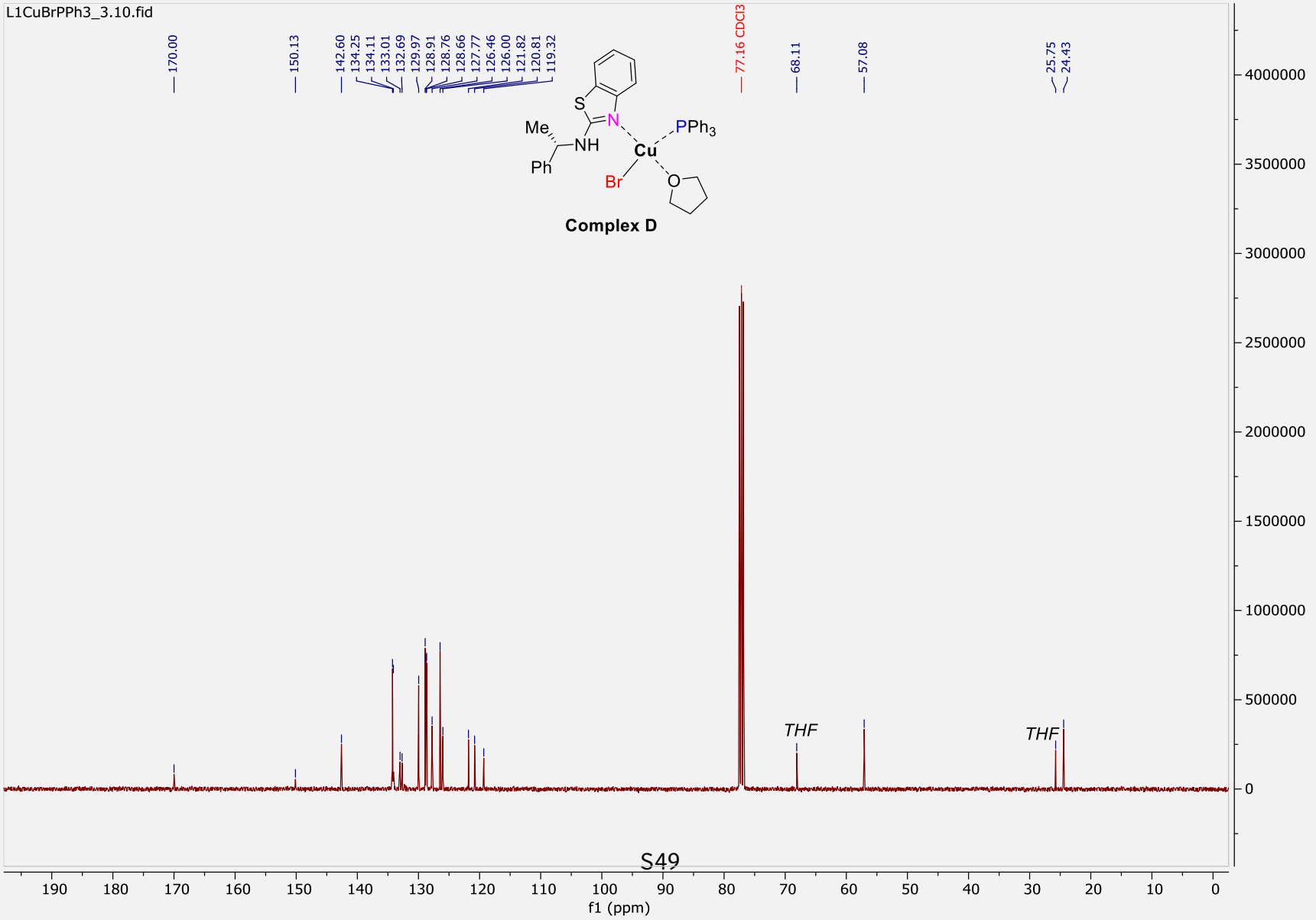

Exp. 50 DMSO_2.10.fid
165.31
152.44
144.43
130.27
128.34
126.82
125.99
125.46
120.87
118.04
53.33
39.52 DMSO
23.15
Me
S
NH
N
7000000
6500000
6000000
5500000
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
-500000
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

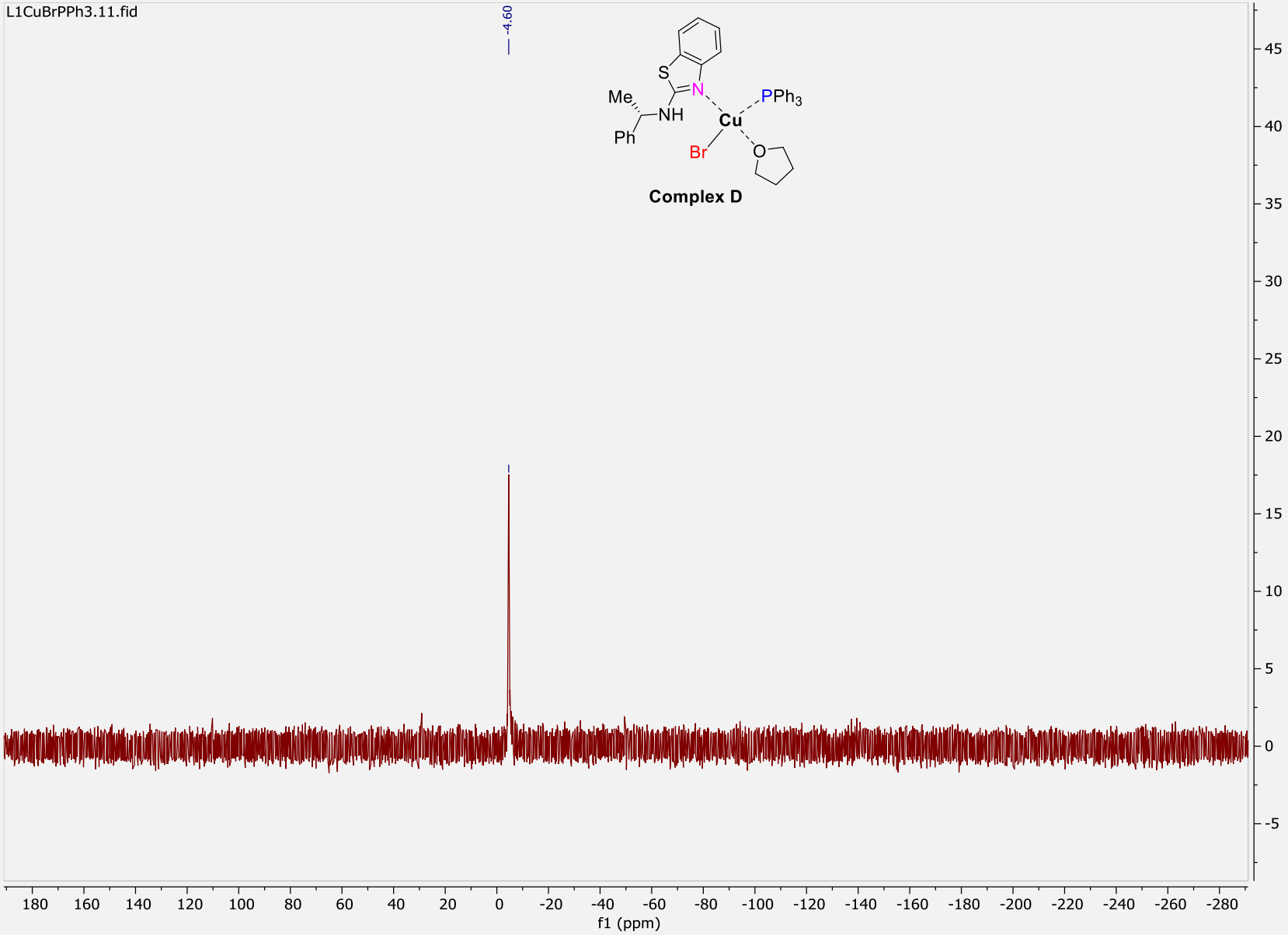

Exp. 51 DMSO.10.fid
Exp. 51
Br
a
S
N
NH
Me
2.50 DMSO
H_2O
N-H
a
1.00
0.93
2.03
3.06
2.01
1.12
3.06
4000
3500
3000
2500
2000
1500
1000
500
0
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

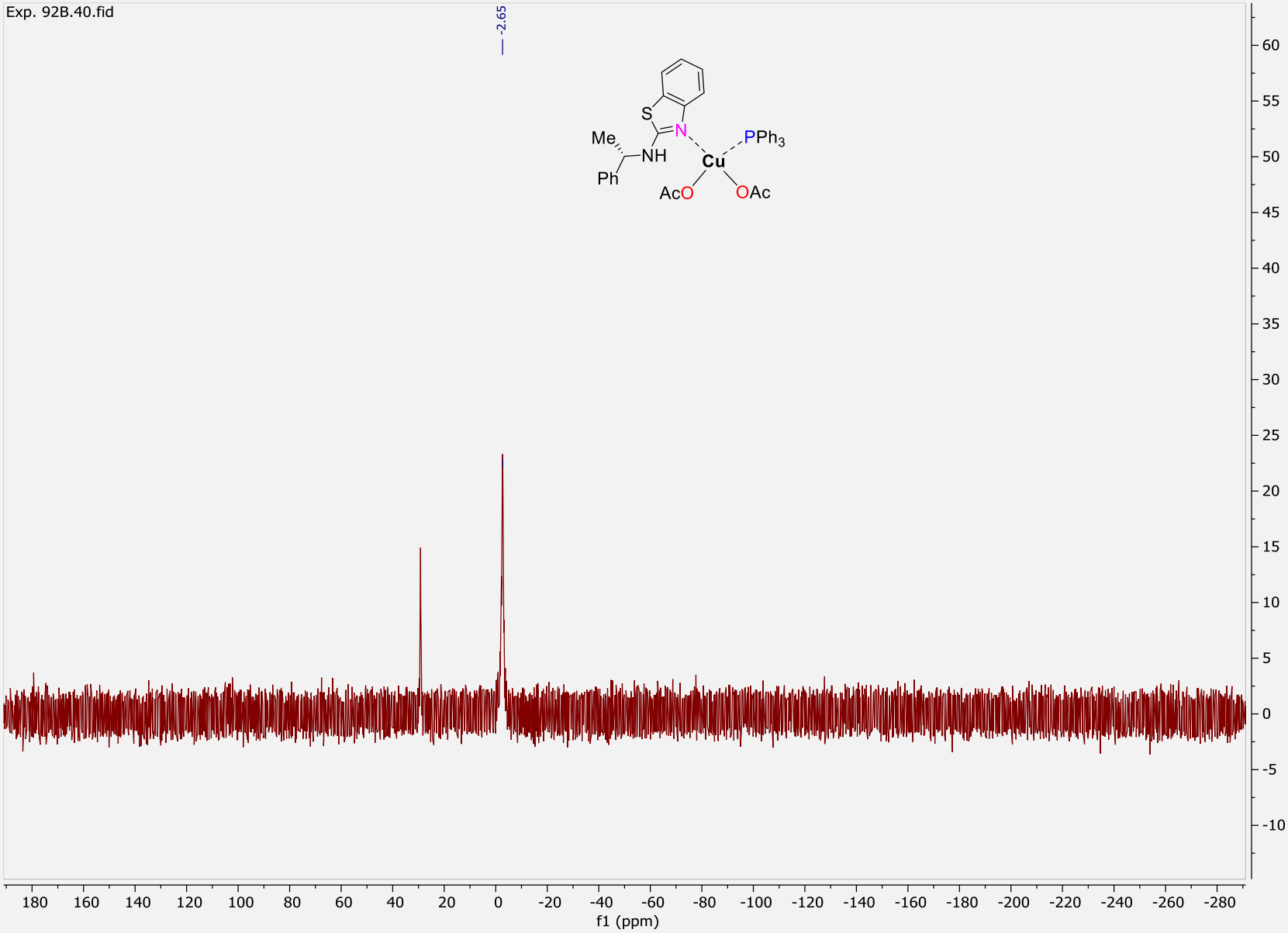

Exp. 51 DMSO_2.10.fid
165.89
151.68
144.16
132.49
128.39
128.34
126.91
125.98
123.31
119.42
112.17
53.40
39.52 DMSO
23.07
Br
S
N
NH
Me
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

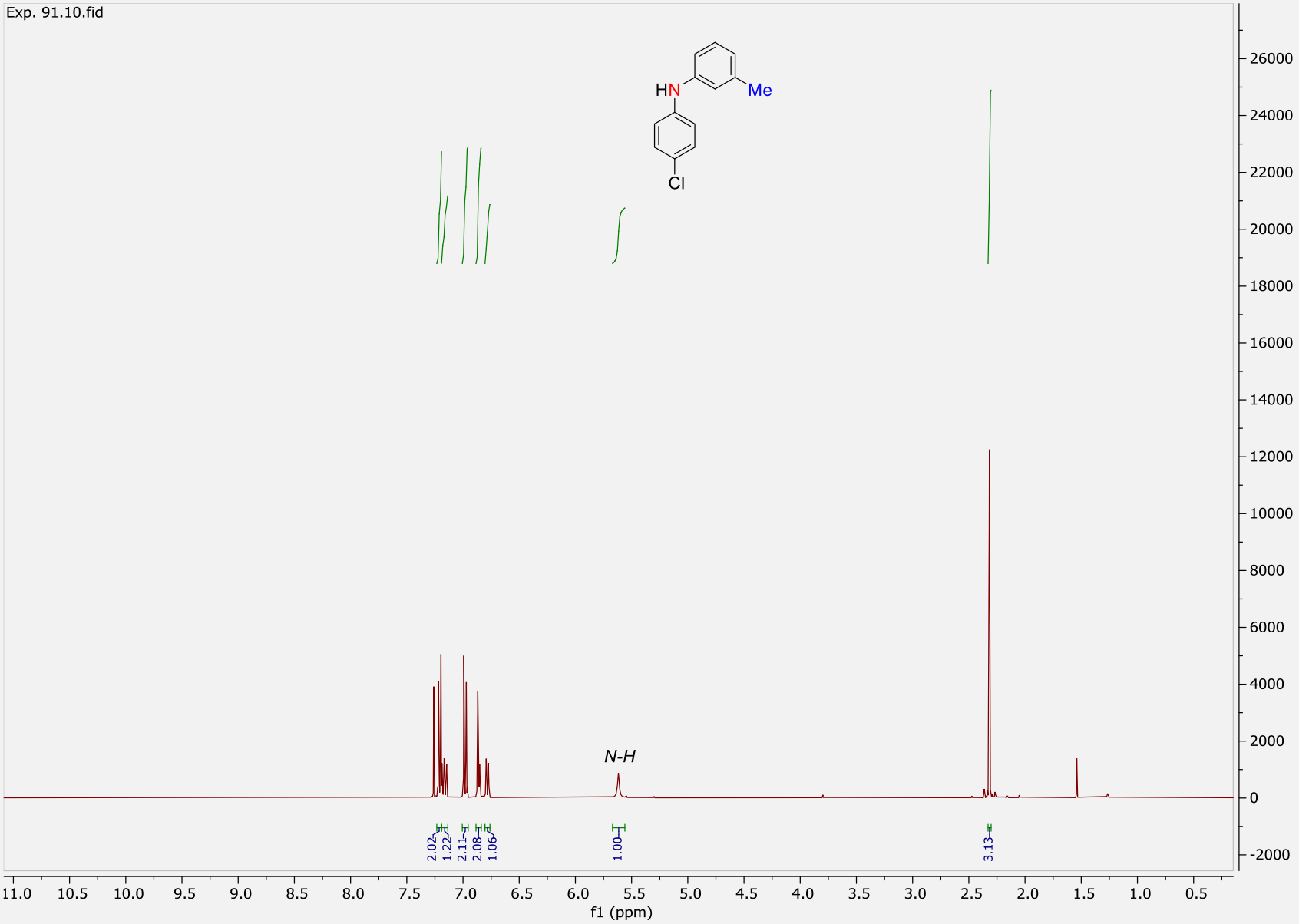

L1CuBrPPh3.10.fid
Complex D
N-H
THF
THF
1.00
6.16
2.99
3.86
9.20
2.12
1.09
3.00
f1 (ppm)

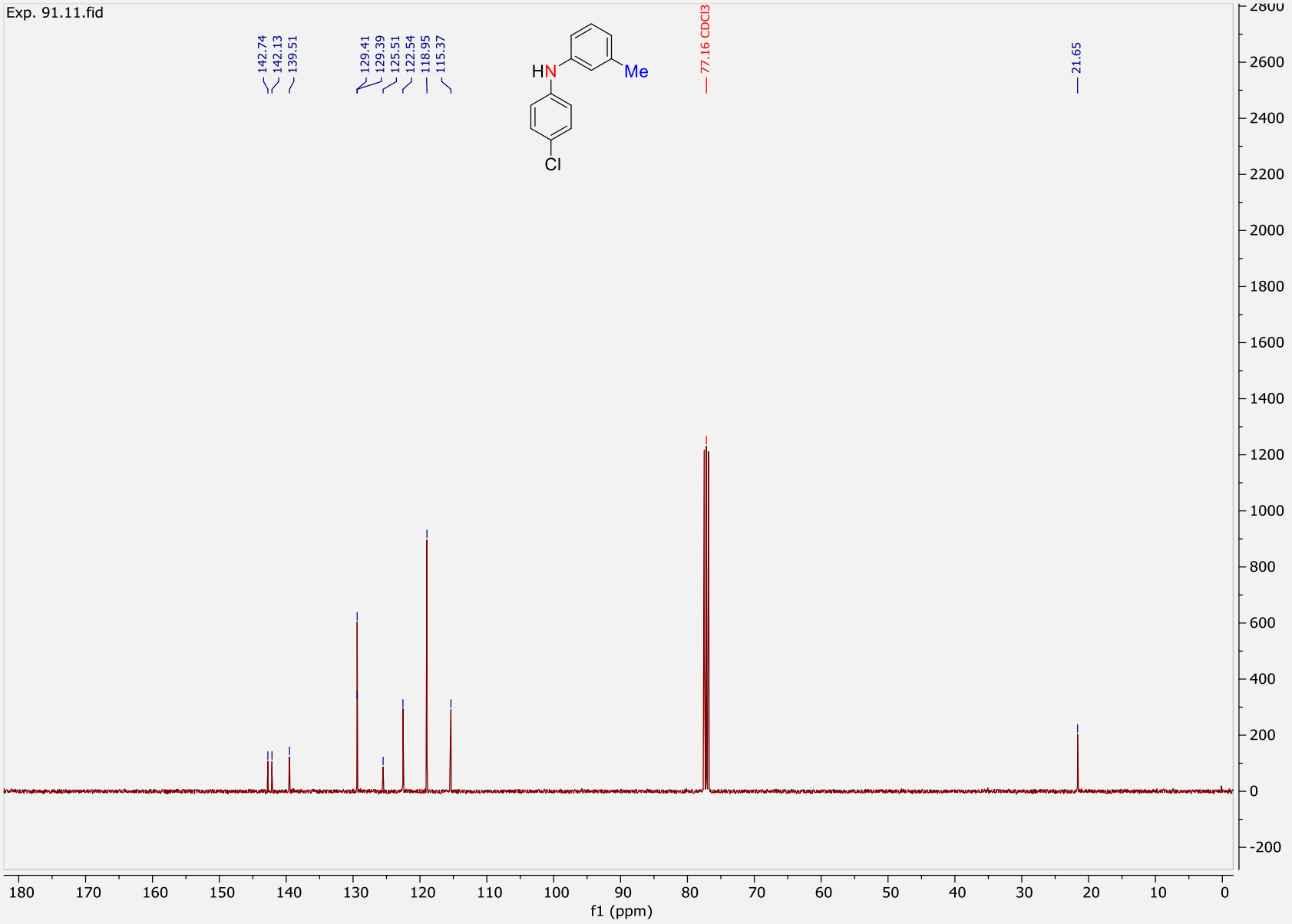
N-H

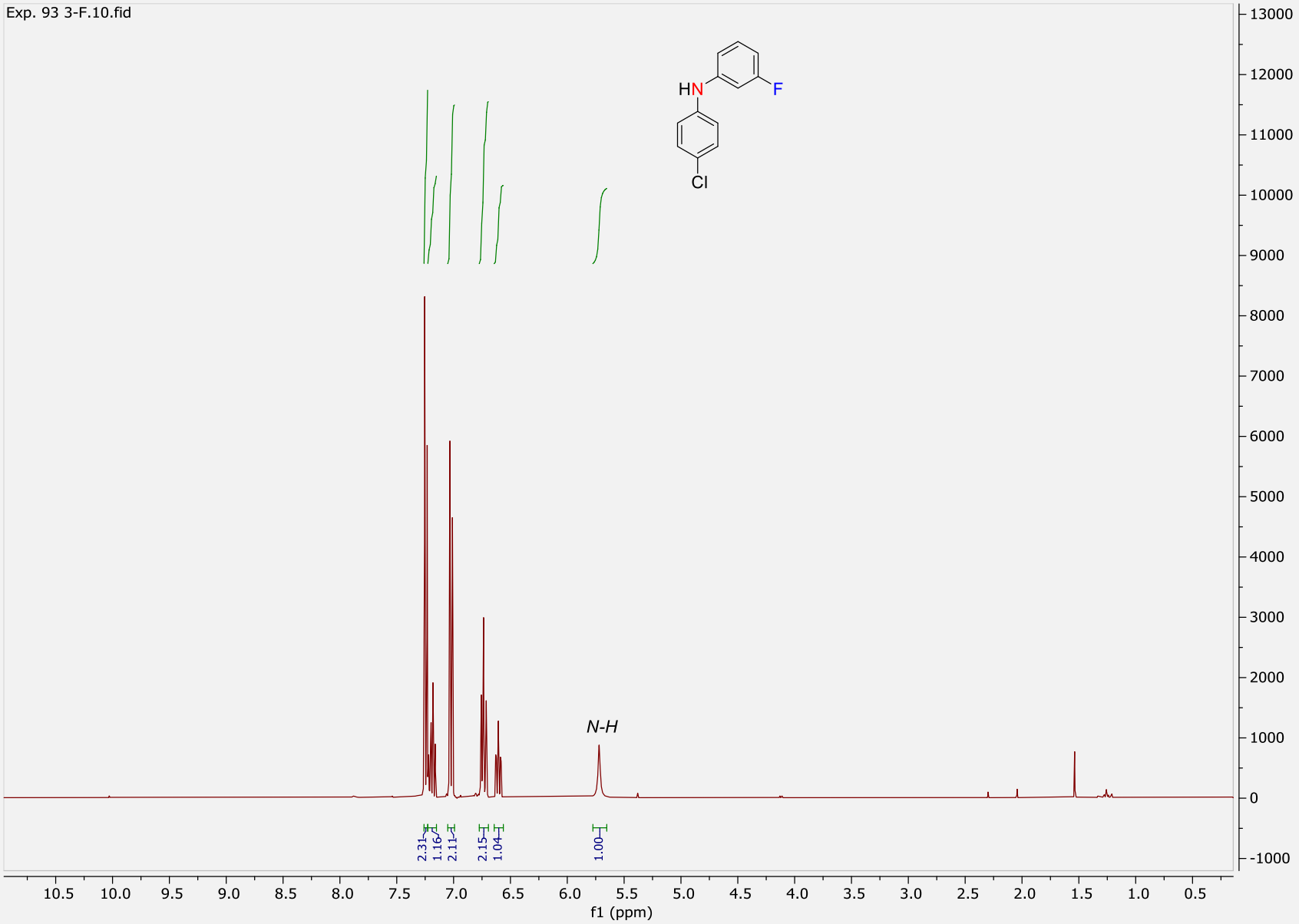

L1CuBrPPh3_3.10.fid
170.00
150.13
142.60
134.25
134.11
133.01
132.69
129.97
128.91
128.76
128.66
127.77
126.46
126.00
121.82
120.81
119.32
77.16 CDCl3
68.11
57.08
25.75
24.43
Me
NH
Ph
S
N
Cu
PPh3
Br
O
Complex D
THF
THF
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

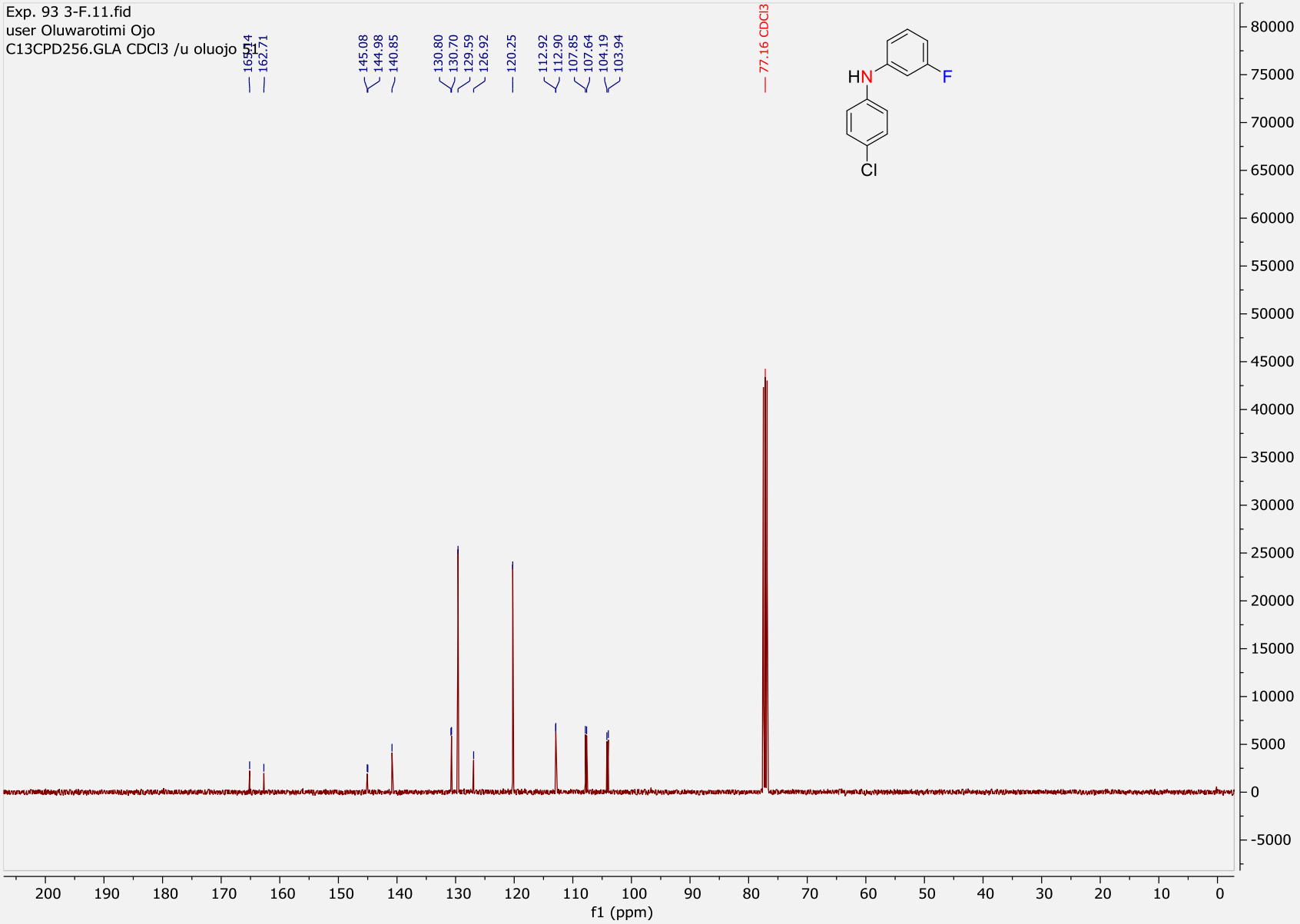

L1CuBrPPh3.11.fid
-4.60
S
N
Me
NH
Ph
Cu
PPh3
Br
O
Complex D
45
40
35
30
25
20
15
10
5
0
-5
180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280
f1 (ppm)

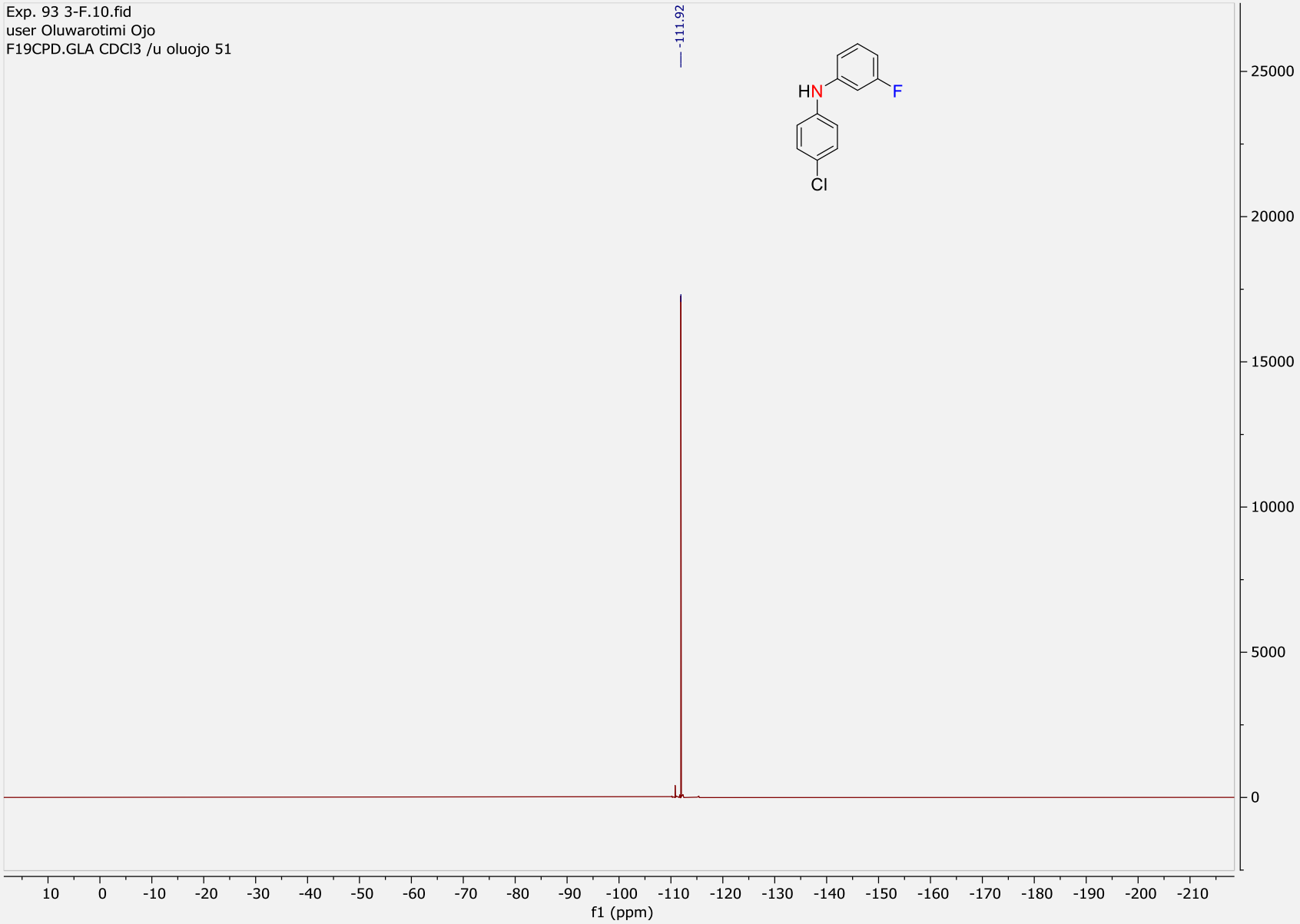

Exp. 92B.40.fid
-2.65
S
N
Me
NH
Ph
Cu
PPh3
AcO
OAc
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280
f1 (ppm)

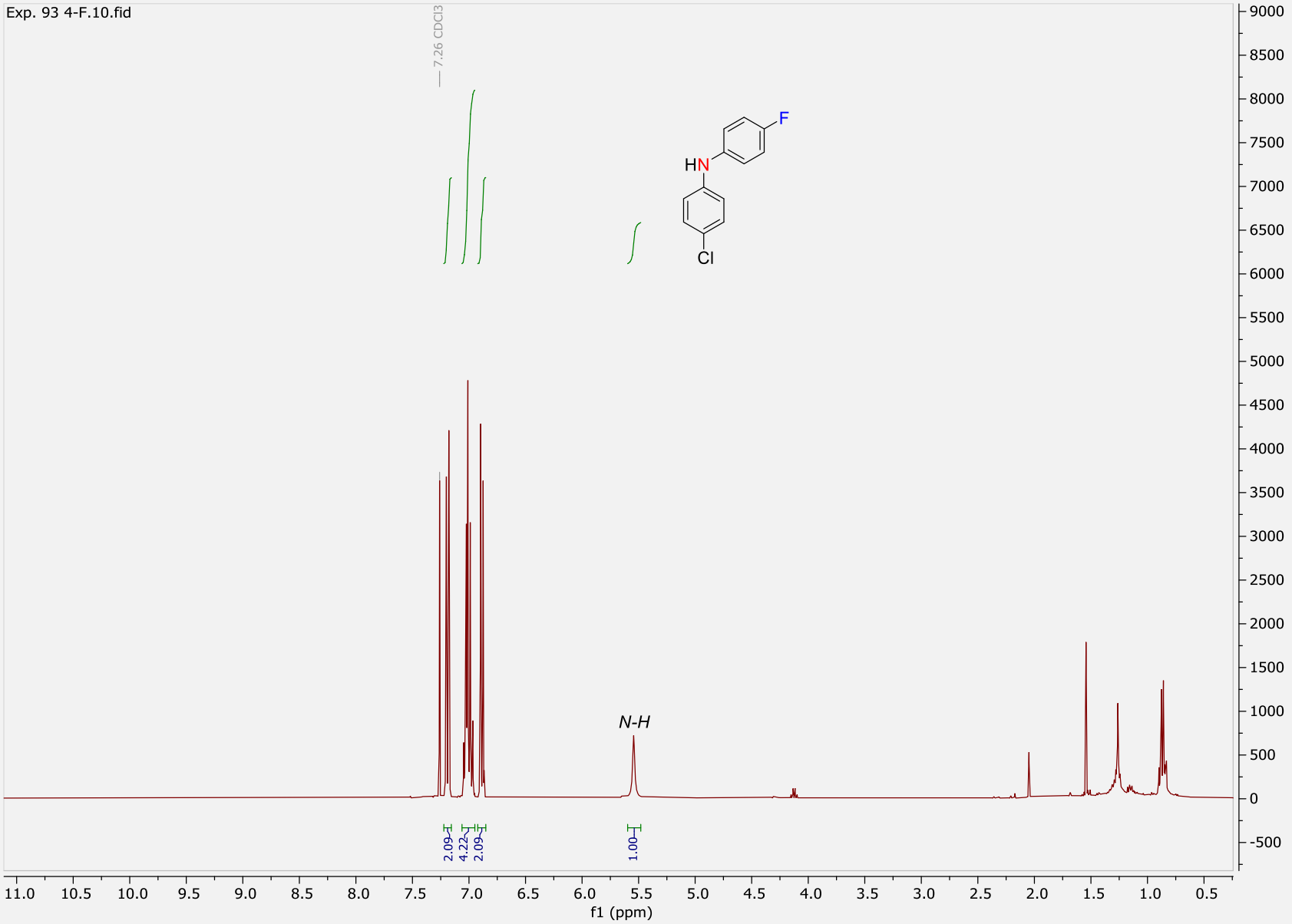

Exp. 91.10.fid
HN
Me
Cl
N-H
2.02
1.22
2.11
2.08
1.06
1.00
3.13
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

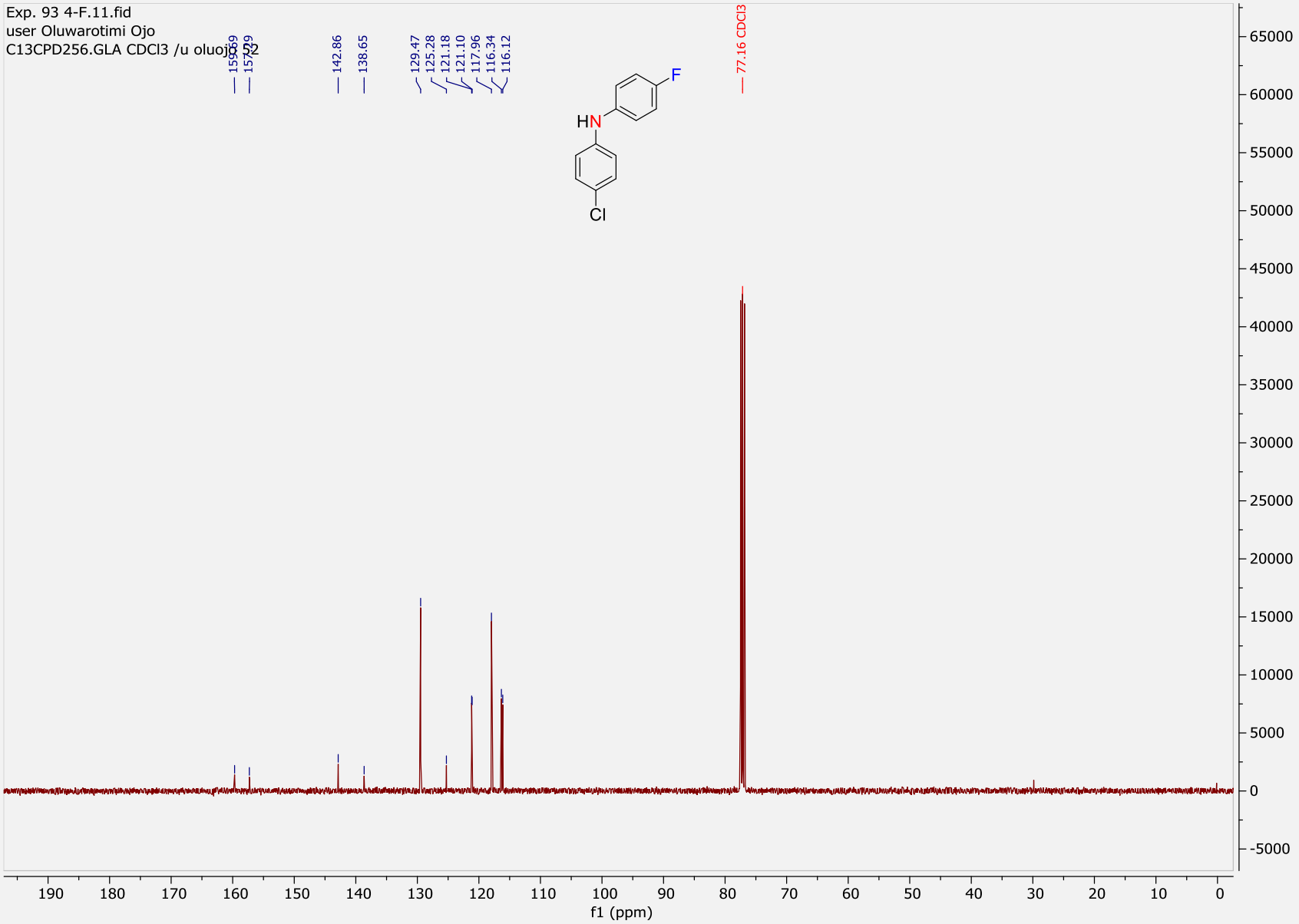

Exp. 91.11.fid
142.74
142.13
139.51
129.41
129.39
125.51
122.54
118.95
115.37
77.16 CDCl3
21.65
HN
Me
Cl
f1 (ppm)

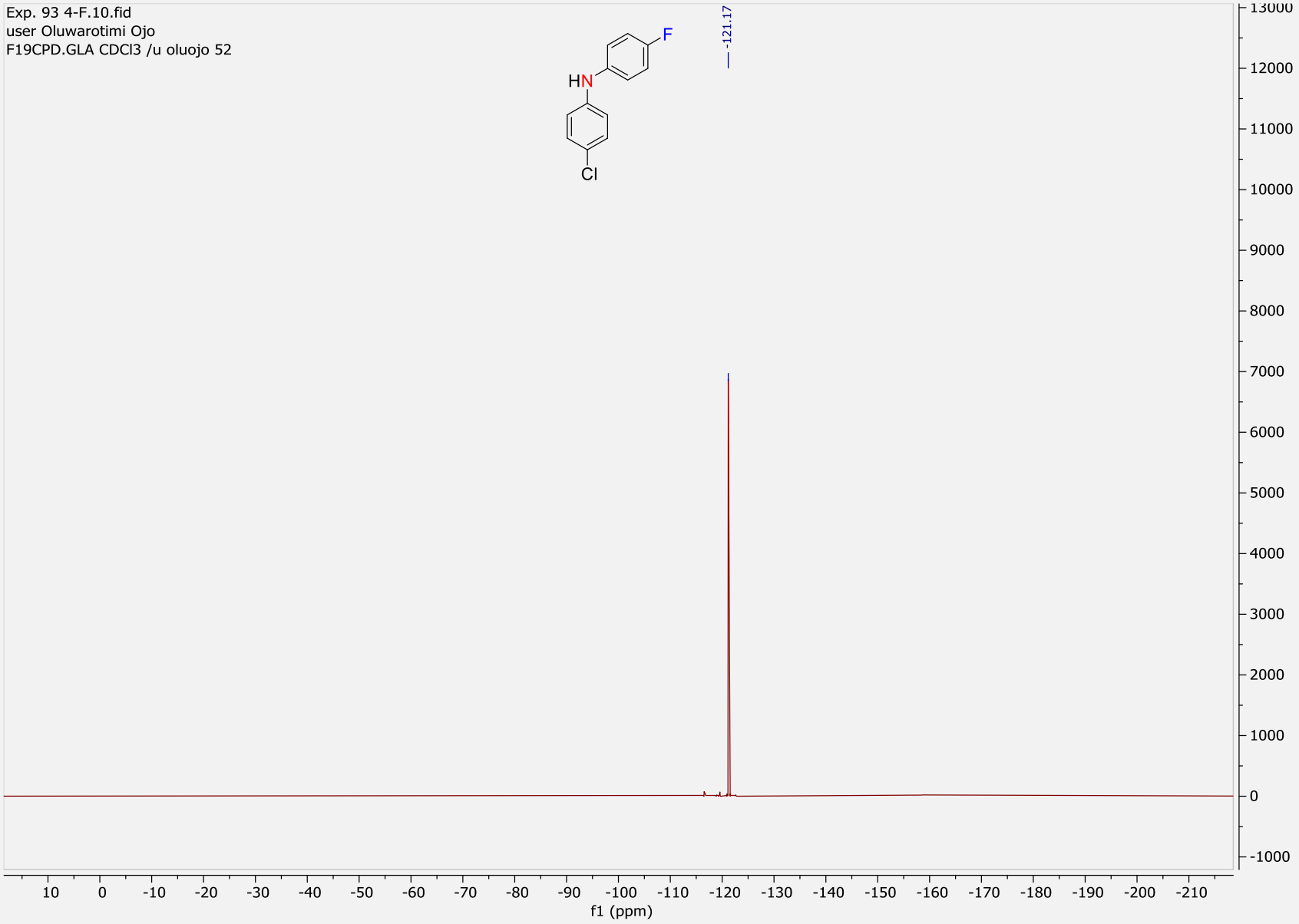

Exp. 93 3-F.10.fid
HN
F
Cl
N-H
2.31
1.16
2.11
2.15
1.04
1.00
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

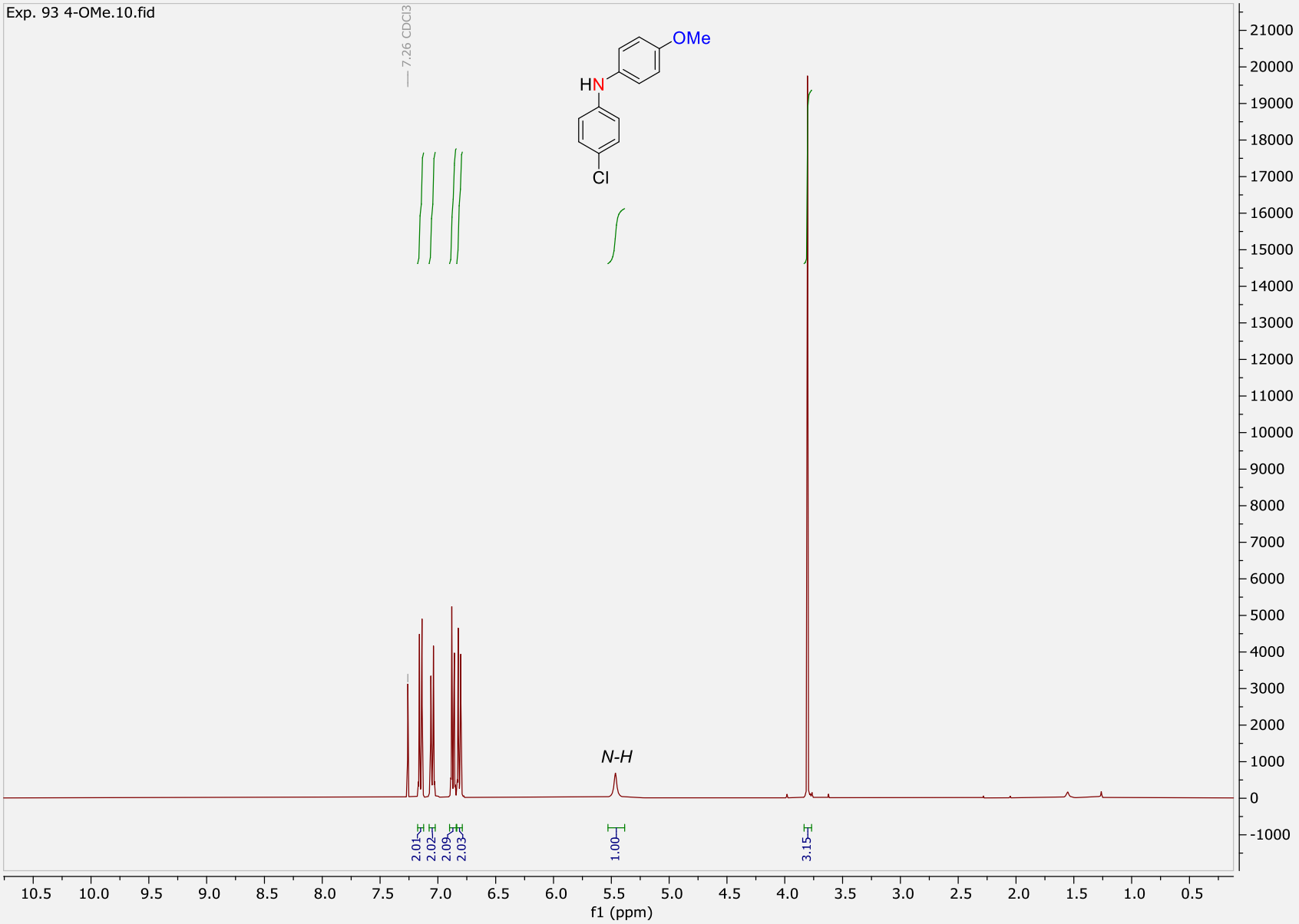

Exp. 93 3-F.11.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 51
165.14
162.71
145.08
144.98
140.85
130.80
130.70
129.59
126.92
120.25
112.92
112.90
107.85
107.64
104.19
103.94
77.16 CDCl3
HN
F
Cl
f1 (ppm)

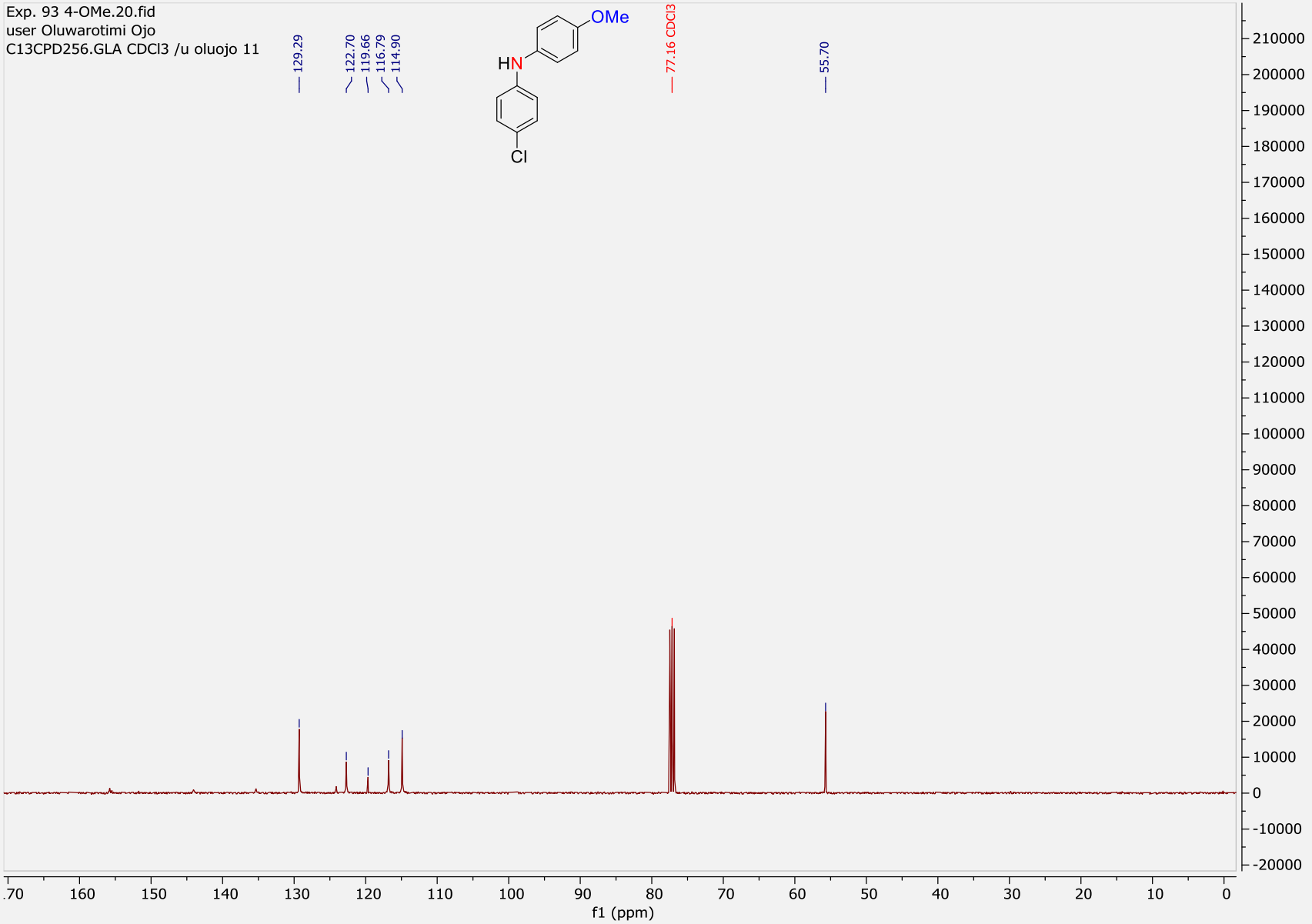

Exp. 93 3-F.10.fid
user Oluwarotimi Ojo
F19CPD.GLA CDCl3 /u oluojo 51
-111.92
HN
F
Cl
25000
20000
15000
10000
5000
0
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

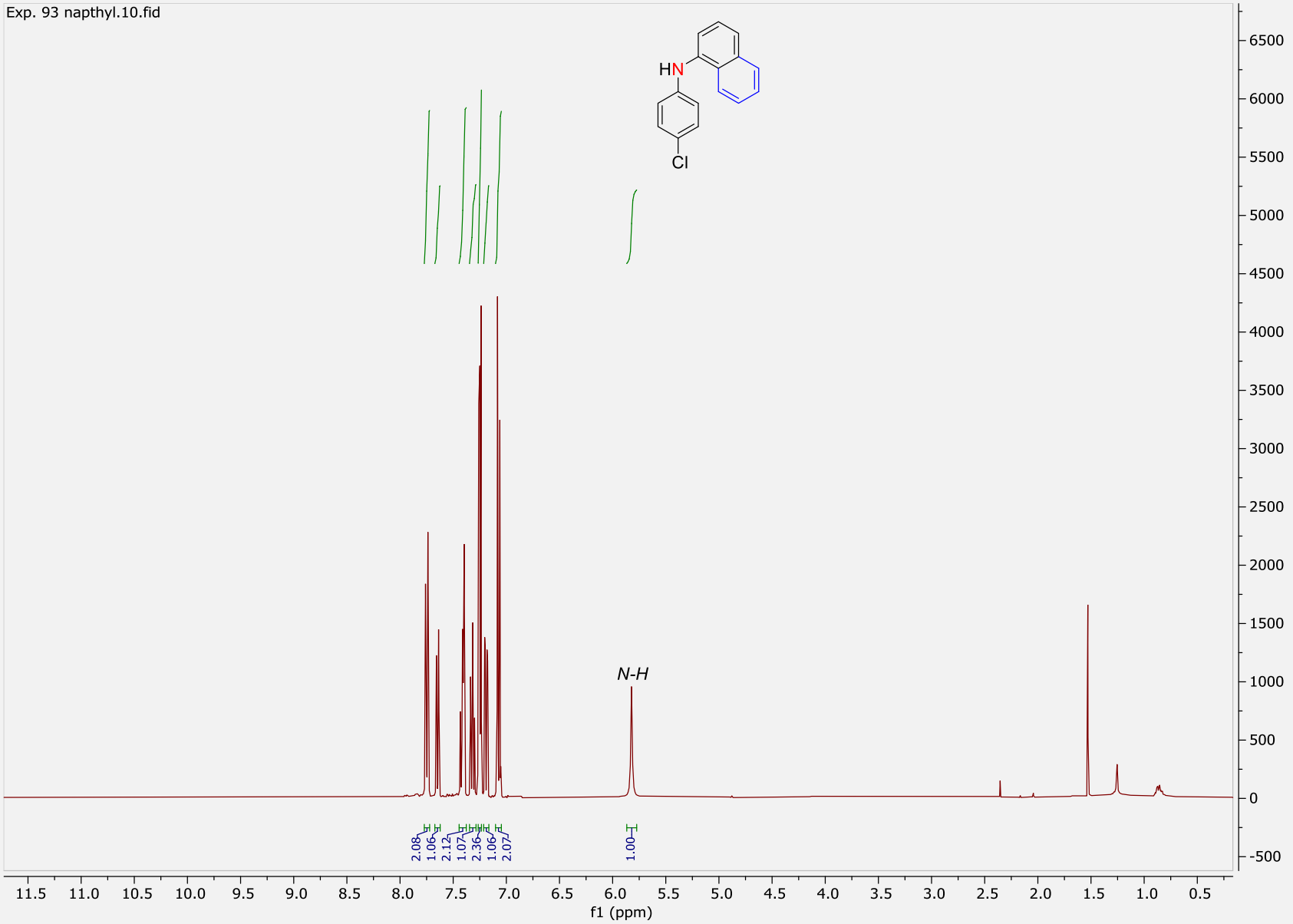

Exp. 93 4-F.10.fid
7.26 CDCl3
F
HN
Cl
N-H
2.09
4.22
2.09
1.00
f1 (ppm)

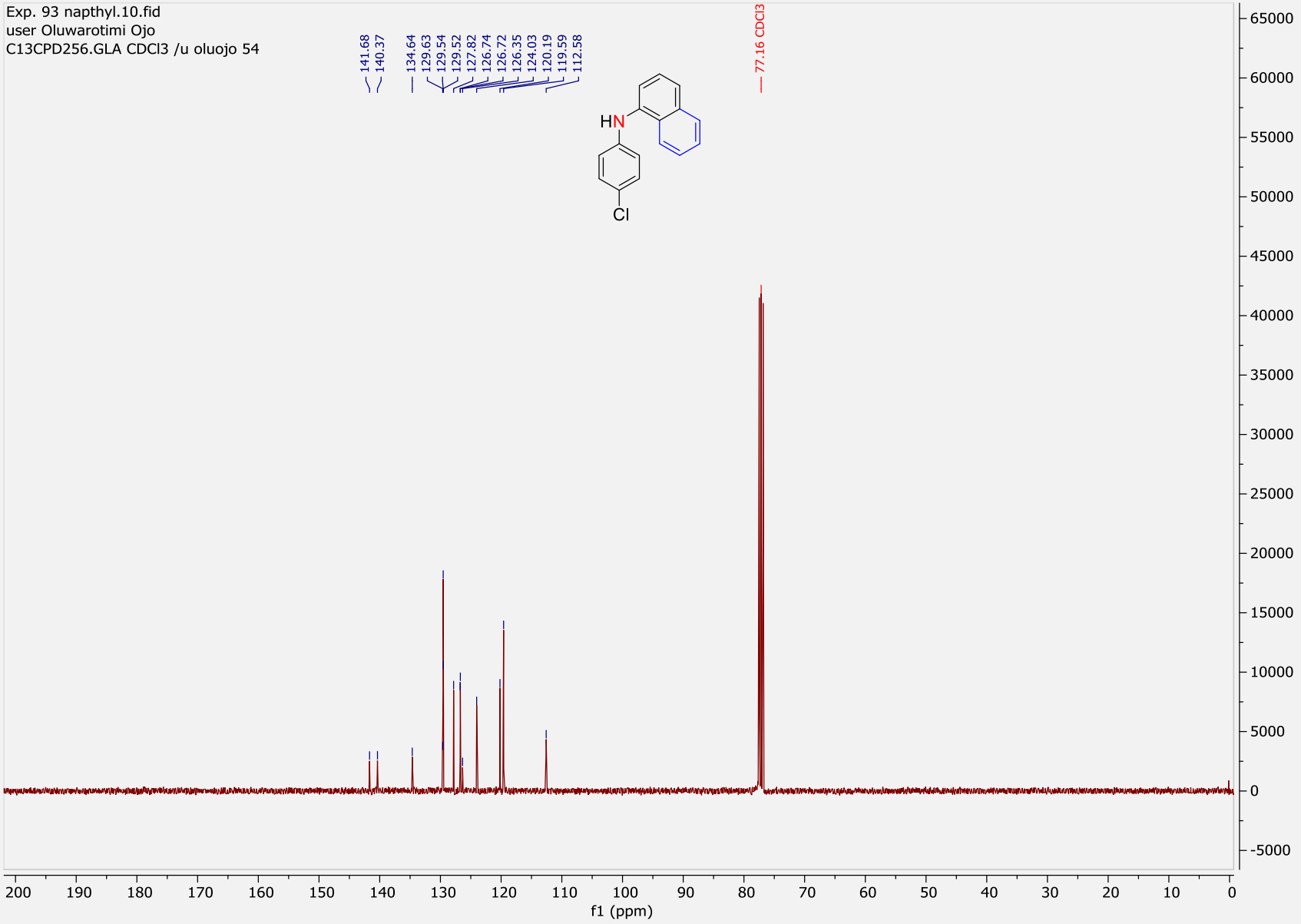

Exp. 93 4-F.11.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 52
159.69
157.29
142.86
138.65
129.47
125.28
121.18
121.10
117.96
116.34
116.12
77.16 CDCl3
HN
F
Cl
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Exp. 93 4-F.10.fid
user Oluwarotimi Ojo
F19CPD.GLA CDCl3 /u oluojo 52
HN
F
Cl
-121.17
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)


Exp. 93 4-OMe.10.fid
7.26 CDCl3
OMe
HN
Cl
N-H
2.01
2.02
2.09
2.03
1.00
3.15
f1 (ppm)


Exp. 93 4-OMe.20.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 11
129.29
122.70
119.66
116.79
114.90
77.16 CDCl3
55.70
OMe
HN
Cl
f1 (ppm)


Exp. 93 napthyl.10.fid
HN
Cl
N-H
2.08
1.06
2.12
1.07
2.36
1.06
2.07
1.00
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


Exp. 93 napthyl.10.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 54
141.68
140.37
134.64
129.63
129.54
129.52
127.82
126.74
126.72
126.35
124.03
120.19
119.59
112.58
77.16 CDCl3
HN
Cl
f1 (ppm)