

Some mechanistic aspects regarding the Suzuki–Miyaura reaction between selected *ortho*-substituted phenylboronic acids and 3,4,5-tribromo-2,6-dimethylpyridine

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Experimental section:

All reactions were carried out in oven-dried pressure tube under argon atmosphere in order to simultaneously exclude oxygen and water. Solvents for the reactions were commercially available and were used without further purification. THF or toluene were dried and distilled by standard methods. All solvents were purchased from Sigma Aldrich. TLC analyses were performed on Merck Kieselgel 60 F-254 plates. The visualization of plates was done under UV light or under iodine vapor. All chemicals employed, including ligands, pre-catalyst, boronic acids and bases, were purchased from commercial sources (Sigma Aldrich and Fluorochem) and used without further purification.

Melting points were determined on an Electrothermal Model IA 9200 apparatus and are uncorrected.

NMR spectra were recorded on a Bruker AVANCE 300 MHz spectrometer operating at 300 MHz for ^1H NMR and 75 MHz for ^{13}C NMR. The spectra were measured in CDCl_3 and are given as δ values (in ppm) relative to TMS. Peak characterization: s = singlet, brs = broad singlet, d = doublet, brd = broad doublet, t = triplet, dd = doublet of doublets, q = quartet, m = multiplet

HRMS spectra were collected on Quattro LC Micromass and LCT Micromass TOF HiRes apparatuses.

X-ray crystallographic data were collected using Xcalibur-R κ -axis single crystal diffractometer from Oxford Diffraction. Data were measured at room temperature using Cu K α monochromatic radiation. After initial corrections the collected data were used to solve crystal structures and then to refine models. SHELXS-97 and SHELXL-97 software was applied for these purposes. Full structural data have been deposited with Cambridge Structural Data Centre under respective deposit numbers.

General procedure of Suzuki–Miyaura reaction:

A mixture of **4** (500 mg, 1.45 mmol), arylboronic acid (1.0–12.0 equiv), palladium catalyst (5.0 mol %), ligand (5.0 mol %) and base (9.0 equiv) in 10 mL of solvent was stirred at required temperature under argon atmosphere for required time. The reaction was monitored by TLC. After completion of the reaction, the mixture was cooled and quenched with cold water (5 mL). The organic phase was extracted with toluene (3 $\times$ 5 mL). Combined organic extracts were dried over anhydrous magnesium sulphate and concentrated under reduced pressure. The crude mixture was purified by flash column chromatography on silica gel (230–400 mesh) using ethyl acetate in hexane to obtain pure compound different-substituted compound in moderate or good yield.

(*aR,aS*)-3,4-Dibromo-5-(2-methoxyphenyl)-2,6-dimethylpyridine (**6**). White solid, yield 30%, mp 69 °C; R_f 0.57 (ethyl acetate : hexane 1:1); ^1H NMR (300 MHz, CDCl_3) δ = 2.23 (s, 3H, CH_3), 2.76 (s, 3H, CH_3), 3.76 (s, 3H, OCH_3), 6.98–7.08 (m, 3H, CH), 7.38–7.44 (m, 1H, CH); ^{13}C NMR (75 MHz, CDCl_3) δ = 23.7, 27.1, 55.8, 111.3, 121.0, 121.7, 128.5, 130.1, 130.5, 134.3, 138.1, 155.9, 156.4, 157.0; HRMS (ESI+) calcd. for $\text{C}_{14}\text{H}_{14}\text{Br}_2\text{NO}$ $[\text{M}+\text{H}]^+$ 371.9422; found 371.9425. **6** * picrate: yellow solid, mp

168.9-169.8 °C, crystallization from CH₂Cl₂/hexane. CCDC-1528450 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

(syn)-(aR,aS)-3-Bromo-4,5-bis(2-methoxyphenyl)-2,6-dimethylpyridine (7). White solid, yield 35%, mp 159 °C; *R*_f 0.45 (ethyl acetate : hexane 1:1); ¹H NMR (300 MHz, CDCl₃) δ=2.29 (s, 3H, CH₃), 2.75 (s, 3H, CH₃), 3.59 (s, 3H, OCH₃), 3.62 (s, 3H, OCH₃), 6.68-6.89 (m, 6H, CH), 7.10-7.19 (m, 2H, CH); ¹H NMR (300 MHz, DMSO) δ= 2.13 (s, 3H, CH₃), 2.64 (s, 3H, CH₃), 3.50 (s, 3H, OCH₃), 3.57 (s, 3H, OCH₃), 6.68-6.92 (m, 6H, CH), 7.09-7.16 (m, 2H, CH); ¹³C NMR (75 MHz, CDCl₃) δ= 23.0, 26.0, 55.0, 55.2, 110.0, 110.3, 119.5, 119.7, 121.0, 126.7, 127.9, 128.7, 129.1, 131.0, 131.6, 132.2, 147.7, 155.0, 155.6, 156.0, 156.5; HRMS (ESI+) calcd. for C₂₁H₂₁BrNO₂ [M+H]⁺ 398.0756; found 398.0739, crystallization from methanol. CCDC-1528451 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

(anti)-(aR,aS)-3-Bromo-4,5-bis(2-methoxyphenyl)-2,6-dimethylpyridine (8). White solid, yield 21%, mp 129.5 °C; *R*_f 0.5 (ethyl acetate : hexane 1:1); ¹H NMR (300 MHz, CDCl₃) δ= 2.24 (s, 3H, CH₃), 2.76 (s, 3H, CH₃), 3.67 (s, 3H, OCH₃), 3.69 (s, 3H, OCH₃), 6.68-6.77 (m, 4H, CH), 6.84-6.91 (m, 2H, CH), 7.08-7.17 (m, 2H, CH); ¹H NMR (300 MHz, DMSO) δ= 2.10 (s, 3H, CH₃), 2.63 (s, 3H, CH₃), 3.64 (s, 6H, OCH₃), 6.72-6.77 (m, 3H, CH), 6.81-6.86 (m, 3H, CH), 7.11-7.17 (m, 2H, CH); ¹³C NMR (75 MHz, CDCl₃) δ= 23.0, 26.0, 55.2, 55.5, 110.1, 110.3, 119.9, 120.1, 121.0, 127.6, 128.9, 129.1, 129.3, 129.3, 130.2, 132.6, 148.3, 155.1, 155.4, 155.9, 156.3; HRMS (ESI+) calcd. for C₂₁H₂₁BrNO₂ [M+H]⁺ 398.0756; found 398.0739.

(anti)-(aR,aS)-4-Bromo-3,5-bis(2-methoxyphenyl)-2,6-dimethylpyridine (9). White solid, yield 21%, mp 187 °C; *R*_f 0.45 (ethyl acetate : hexane 1:1); ¹H NMR (300 MHz, CDCl₃) δ= 2.32 (s, 6H, CH₃), 3.78 (s, 6H, OCH₃), 6.98-7.07 (m, 4H, CH), 7.14-7.17 (m, 2H, CH), 7.37-7.43 (m, 2H, CH); ¹³C NMR (75 MHz, CDCl₃) δ= 23.8, 55.6, 111.1, 120.7, 128.5, 129.5, 130.9, 132.2, 156.1, 156.5; HRMS (ESI+) calcd. for C₂₁H₂₁BrNO₂ [M+H]⁺ 398.0756; found 398.0739, crystallization from methanol. CCDC-1528454 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

(syn)-4-Bromo-3,5-bis(2-methoxyphenyl)-2,6-dimethylpyridine (10). White solid, mp 130 °C; *R*_f 0.45 (ethyl acetate : hexane 1:1); ¹H NMR (300 MHz, CDCl₃) δ= 2.33 (s, 6H, CH₃), 3.78 (s, 6H, OCH₃), 6.98-7.17 (m, 6H, CH), 7.37-7.43 (m, 2H, CH); ¹³C NMR (75 MHz, CDCl₃) δ= 23.8, 55.7, 111.3, 120.7, 128.5, 129.5, 130.8, 132.3, 156.1, 156.6; HRMS (ESI+) calcd. for C₂₁H₂₁BrNO₂ [M+H]⁺ 398.0756; found

398.0739. **10** * picrate: yellow solid, mp 206.8-207.9°C, crystallization from dichloromethane/hexane. CCDC-1528457 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

(*anti, syn*)-3,4,5-Tris(2-chlorophenyl)-2,6-dimethylpyridine (**12**). White solid, yield 45%, mp 204.5 °C; R_f 0.36 (20% ethyl acetate in hexane); ^1H NMR (300 MHz, CDCl_3) δ = 2.34 (s, 6H, CH_3), 6.84-6.89 (m, 2H, CH), 6.95-6.98 (m, 1H, CH), 7.07-7.14 (m, 4H, CH), 7.22-7.27 (m, 3H, CH), 7.34-7.38 (m, 2H, CH); ^{13}C NMR (75 MHz, CDCl_3) δ = 23.2, 125.7, 126.4, 128.1, 128.8, 129.0, 129.0, 130.2, 130.8, 131.9, 133.5, 136.9, 137.1, 155.8; HRMS (ESI+) calcd. for $\text{C}_{25}\text{H}_{19}\text{Cl}_3\text{N}$ $[\text{M}+\text{H}]^+$ 438.0583; found 438.0579. **12** * HCl: white solid, mp 233.5-235°C, crystallization from methanol/ethyl acetate CCDC-1528707 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

(*anti, anti*)-(a*R*,a*S*)-3,4,5-Tris(2-chlorophenyl)-2,6-dimethylpyridine (**13**). White solid, yield 39%, mp 226 °C; R_f 0.36 (20% ethyl acetate in hexane); ^1H NMR (300 MHz, CDCl_3) δ = 2.37 (s, 3H, CH_3), 2.45 (s, 3H, CH_3), 6.86-6.91 (m, 3H, CH), 6.93-6.98 (m, 2H, CH), 7.06-7.14 (m, 4H, CH), 7.25-7.29 (m, 2H, CH), 7.34-7.37 (m, 1H, CH); ^{13}C NMR (75 MHz, CDCl_3) δ = 23.2, 125.2, 125.8, 126.6, 128.5, 128.6, 128.9, 129.6, 130.6, 130.7, 130.8, 131.0, 132.7, 133.4, 134.4, 136.2, 136.6, 137.4, 146.3, 155.8, 156.1; HRMS (ESI+) calcd. for $\text{C}_{25}\text{H}_{19}\text{Cl}_3\text{N}$ $[\text{M}+\text{H}]^+$ 438.0583; found 438.0579. **13** * HCl: white solid, mp 254-255.5°C, crystallization from dichloromethane/heptane. CCDC-1528679 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

(*syn, syn*)-3,4,5-Tris(2-chlorophenyl)-2,6-dimethylpyridine (**14**). White solid, yield 15%, mp 108 °C; R_f 0.17 (20% ethyl acetate in hexane); ^1H NMR (300 MHz, CDCl_3) δ = 2.42 (s, 6H, CH), 6.86-7.13 (m, 10H, CH), 7.30-7.33 (m, 2H, CH); ^{13}C NMR (75 MHz, CDCl_3) δ = 23.5, 124.6, 125.9, 127.7, 128.5, 128.8, 129.5, 129.8, 131.3, 132.7, 133.3, 133.8, 134.8, 135.7, 136.6, 146.7, 156.2; HRMS (ESI+) calcd. for $\text{C}_{25}\text{H}_{19}\text{Cl}_3\text{N}$ $[\text{M}+\text{H}]^+$ 438.0583; found 438.0579, crystallization from ethyl acetate. CCDC-1528721 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

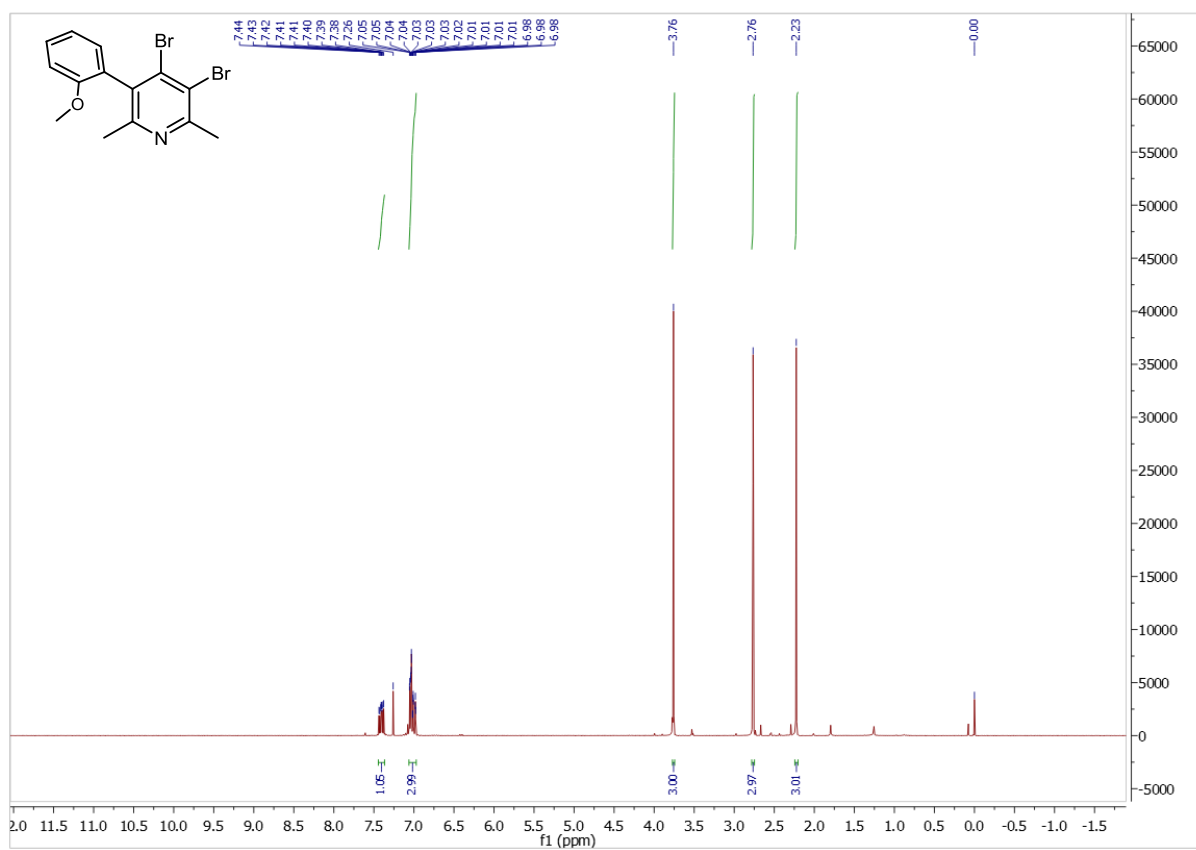
(*anti*)-(a*R*,a*S*)-3-Bromo-4,5-bis(2-chlorophenyl)-2,6-dimethylpyridine (**15**). White solid, yield 48%, mp 110.5 °C; R_f 0.42 (20% ethyl acetate in hexane); ^1H NMR (300 MHz, CDCl_3) δ = 2.62 (s, 3H, CH), 2.79 (s, 6H, CH), 7.07-7.18 (m, 6H, CH), 7.26-7.28 (m, 1H, CH), 7.29-7.30 (m, 1H, CH); ^{13}C NMR (75 MHz, CDCl_3) δ = 22.7, 25.8, 120.2,

126.6, 126.8, 128.9, 128.9, 129.0, 129.3, 126.4, 129.9, 132.4, 132.5, 133.3, 136.8, 138.0, 147.6, 155.0, 156.6; HRMS (ESI+) calcd. for C₁₉H₁₅BrCl₂N [M+H]⁺ 407.9742; found 407.9748. **15** * picrate: yellow solid, mp 214-216°C, crystallization from dichloromethane/methanol. CCDC-1528655 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

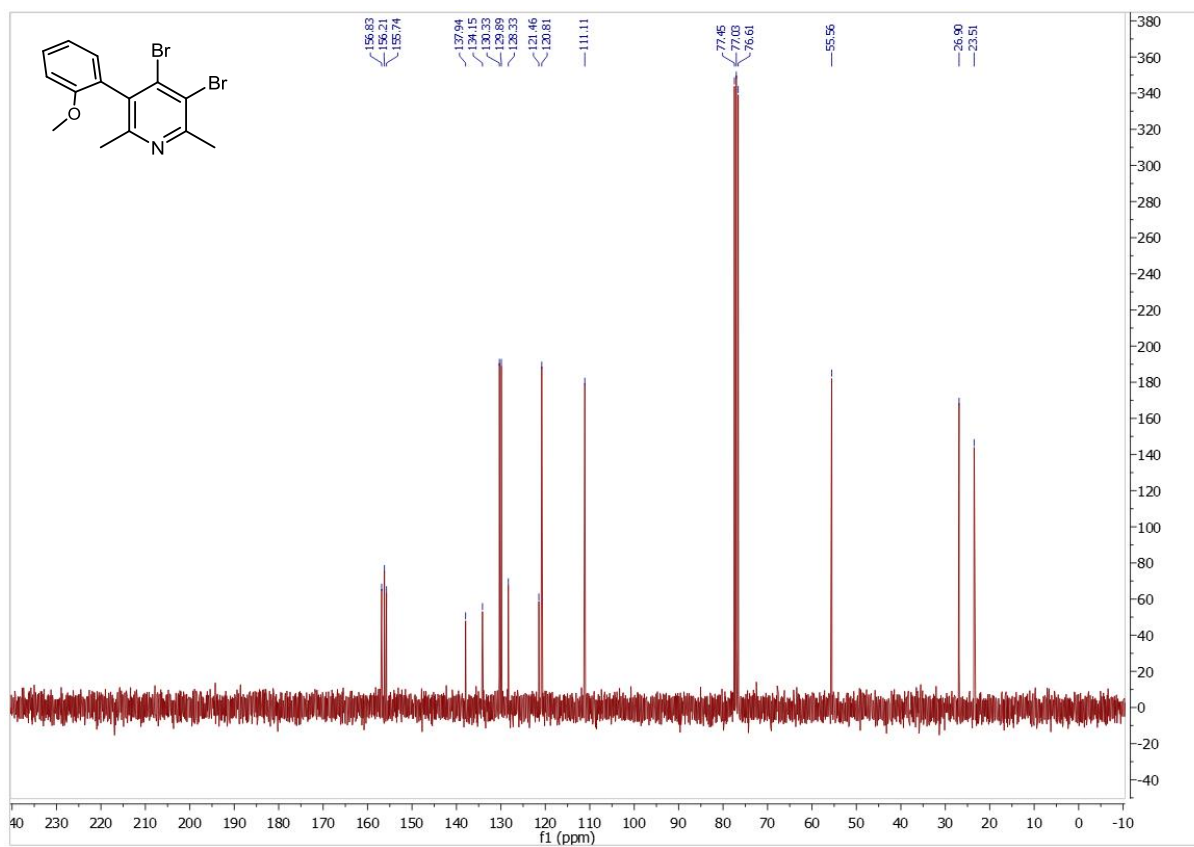
(syn)-(aR,aS)-3-Bromo-4,5-bis(2-chlorophenyl)-2,6-dimethylpyridine (16). White solid, yield 13%, mp 154 °C; R_f 0.404 (20% ethyl acetate in hexane); ¹H NMR (300 MHz, CDCl₃) δ= 2.29 (s, 3H, CH₃), 2.80 (s, 3H, CH₃), 6.97-7.15 (m, 6H, CH), 7.25-7.31 (m, 2H, CH); ¹³C NMR (75 MHz, CDCl₃) δ= 23.1, 26.2, 120.8, 125.9, 126.2, 129.2, 129.4, 129.7, 129.9, 131.5, 132.4, 132.7, 133.3, 134.3, 136.1, 137.4, 147.9, 155.4, 156.8; HRMS (ESI+) calcd. for C₁₉H₁₅BrCl₂N [M+H]⁺ 407.9742; found 407.9748, crystallization from ethyl acetate. CCDC-1528653 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

(anti)-(aR,aS)-4-Bromo-3,5-bis(2-chlorophenyl)-2,6-dimethylpyridine (17). White solid, yield 18%, mp 229 °C; R_f 0.34 (20% ethyl acetate in hexane); ¹H NMR (300 MHz, CDCl₃) δ= 2.34 (s, 6H, CH₃), 7.24-7.28 (m, 2H, CH), 7.35-7.41 (m, 4H, CH), 7.49-7.54 (m, 2H, CH); ¹³C NMR (75 MHz, CDCl₃) δ= 23.7, 127.2, 129.6, 129.7, 131.1, 133.1, 133.5, 136.5, 138.0, 156.5; HRMS (ESI+) calcd. for C₁₉H₁₅BrCl₂N [M+H]⁺ 407.9742; found 407.9748, crystallization from ethyl acetate. CCDC-1528652 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

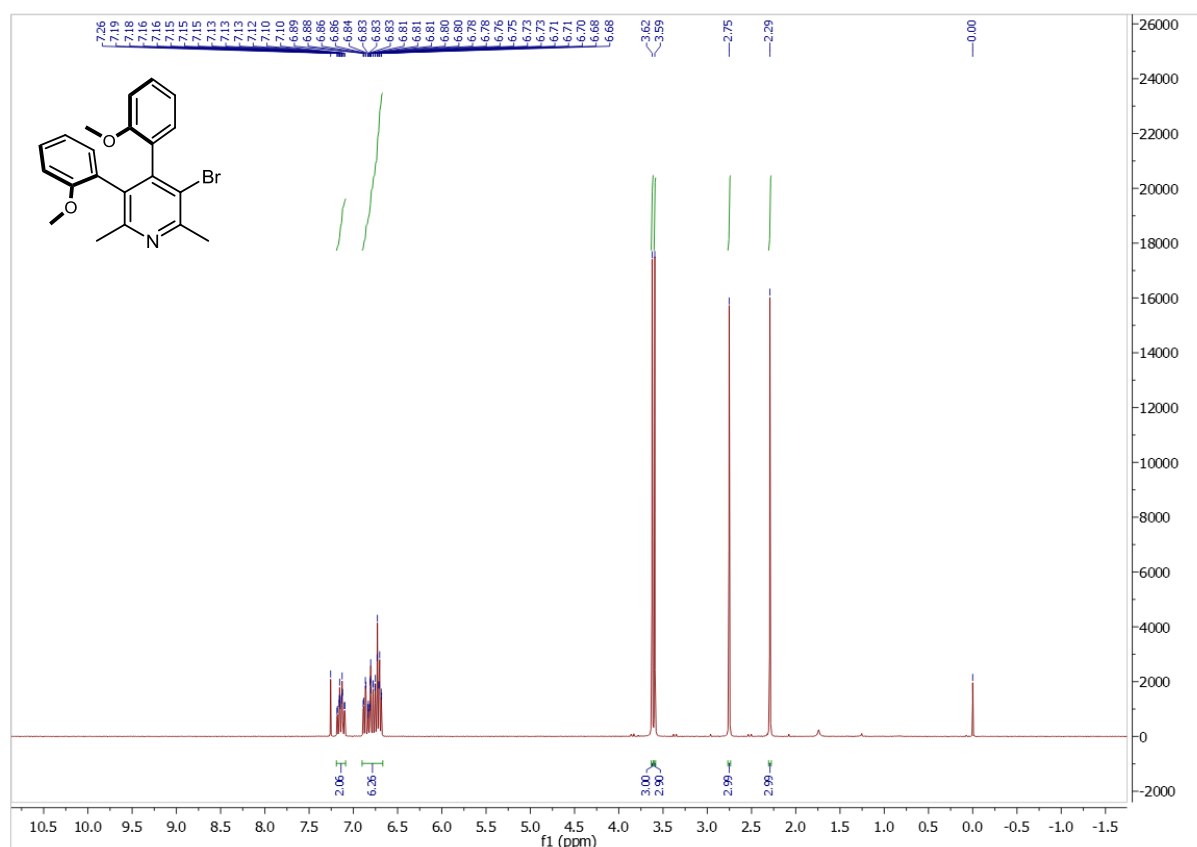
^1H NMR spectrum of **6** (300 MHz, CDCl_3)



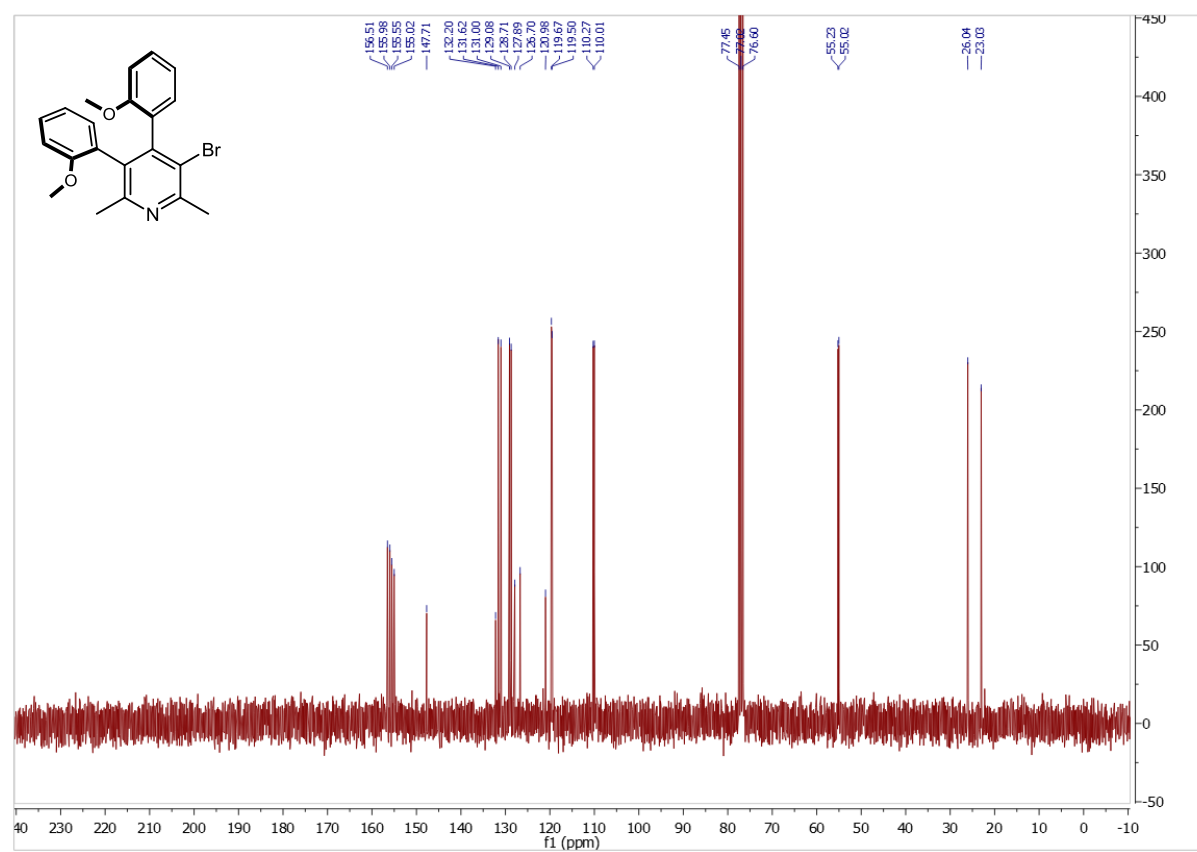
^{13}C NMR spectrum of **6** (75 MHz, CDCl_3)

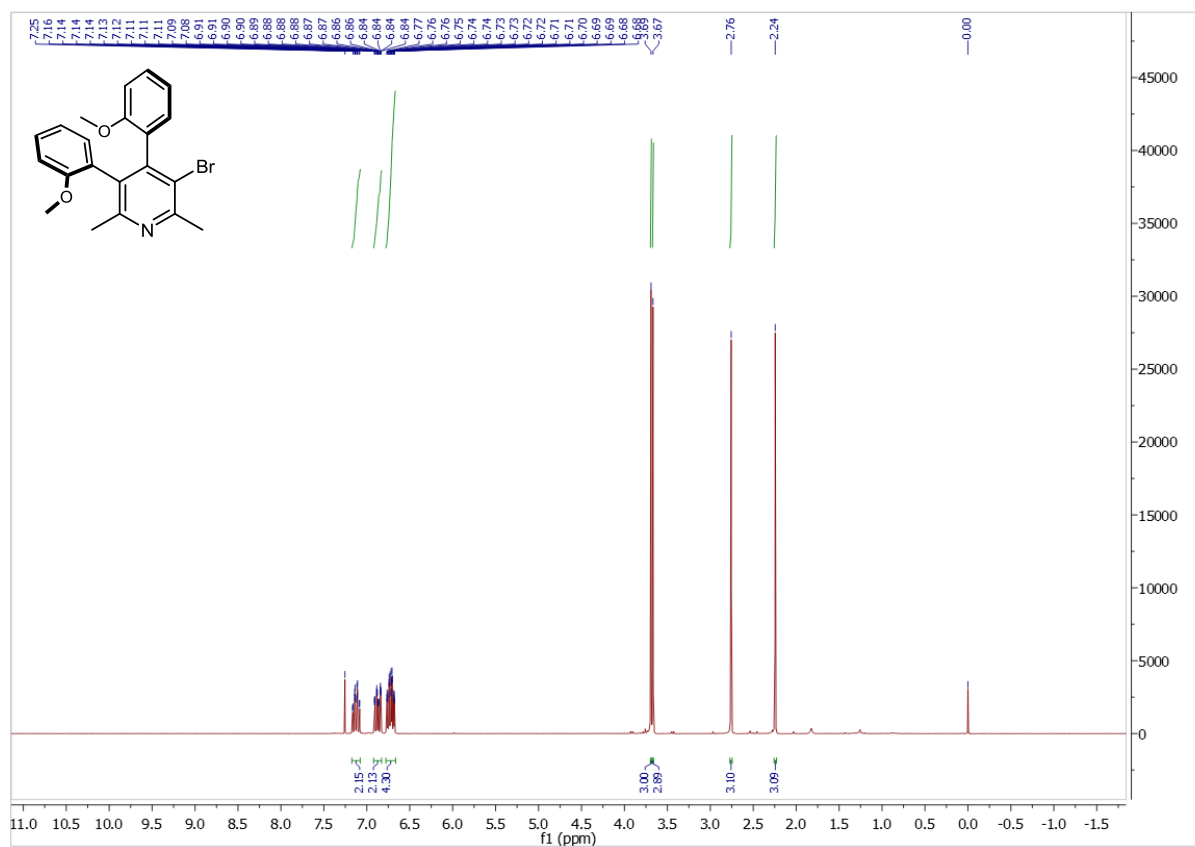


^1H NMR spectrum of **7** (300 MHz, CDCl_3)

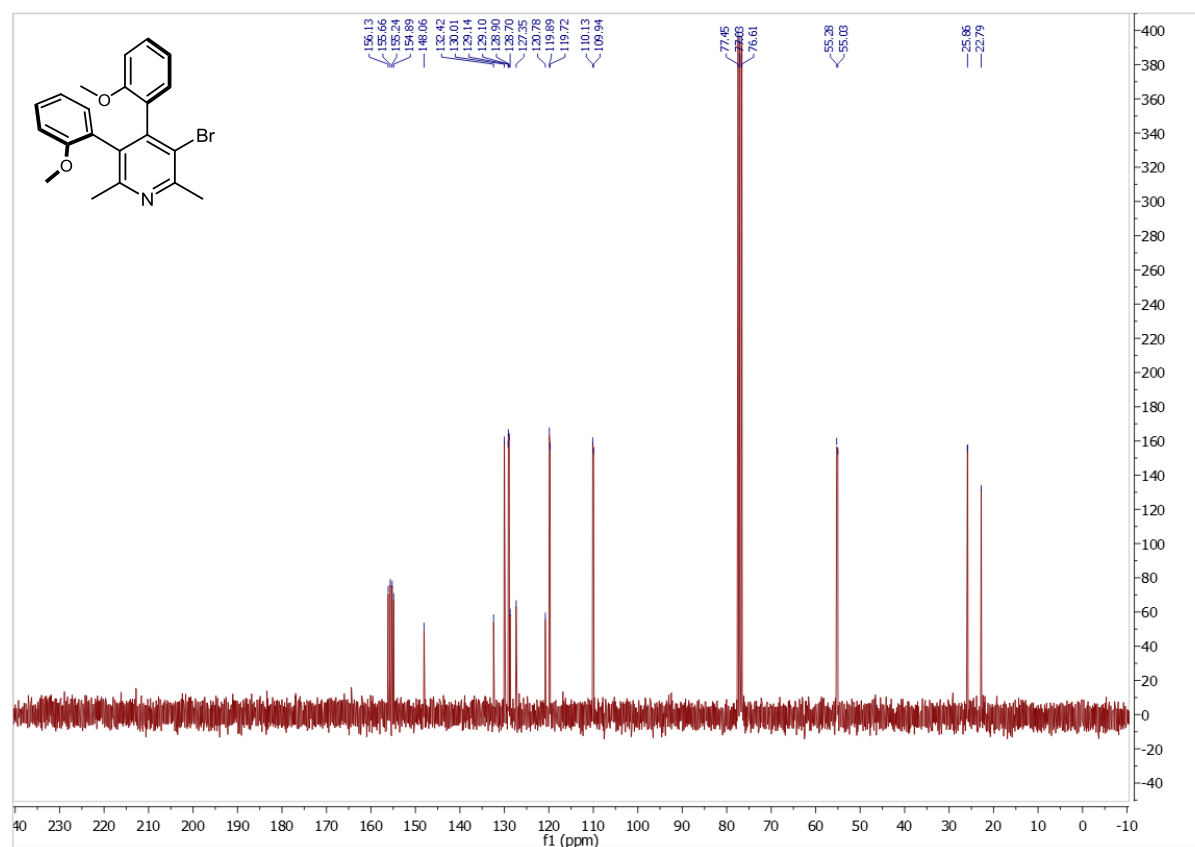


^{13}C NMR spectrum of **7** (75 MHz, CDCl_3)

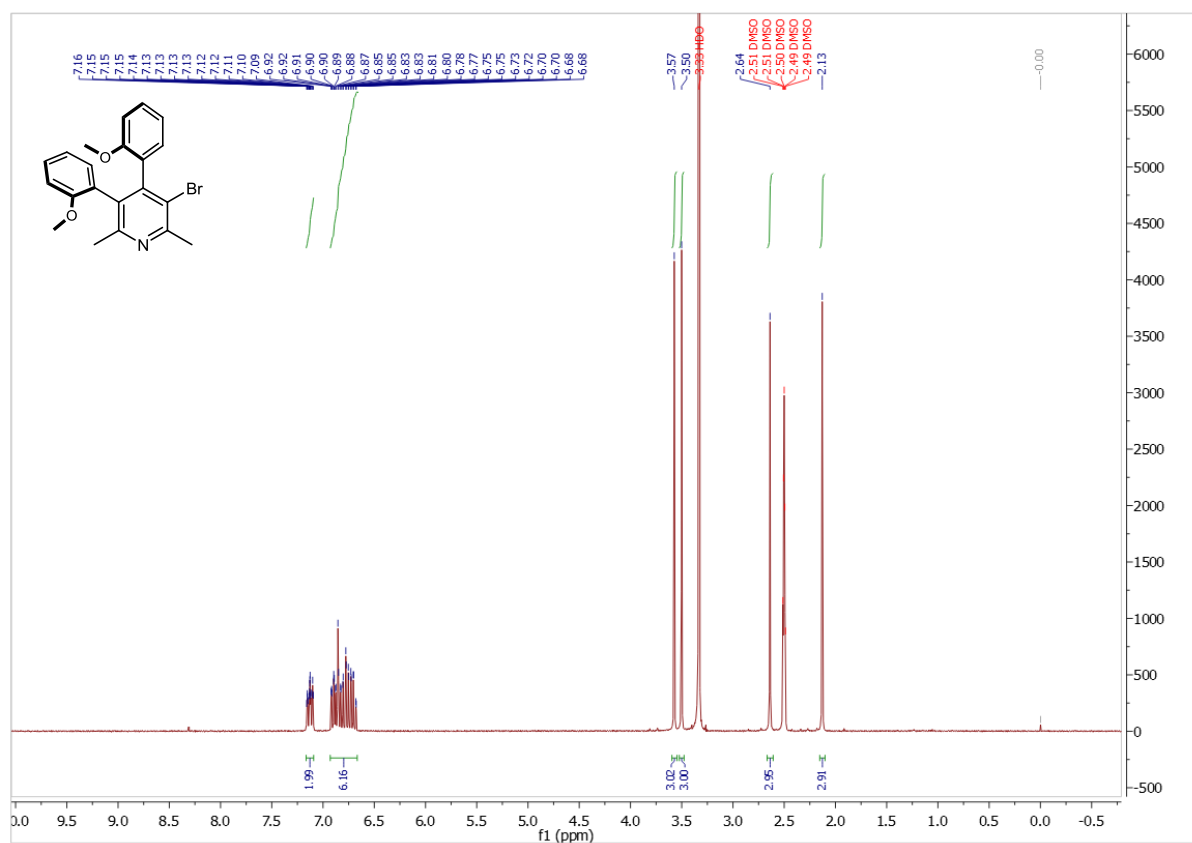


¹H NMR spectrum of **8** (300 MHz, CDCl₃)

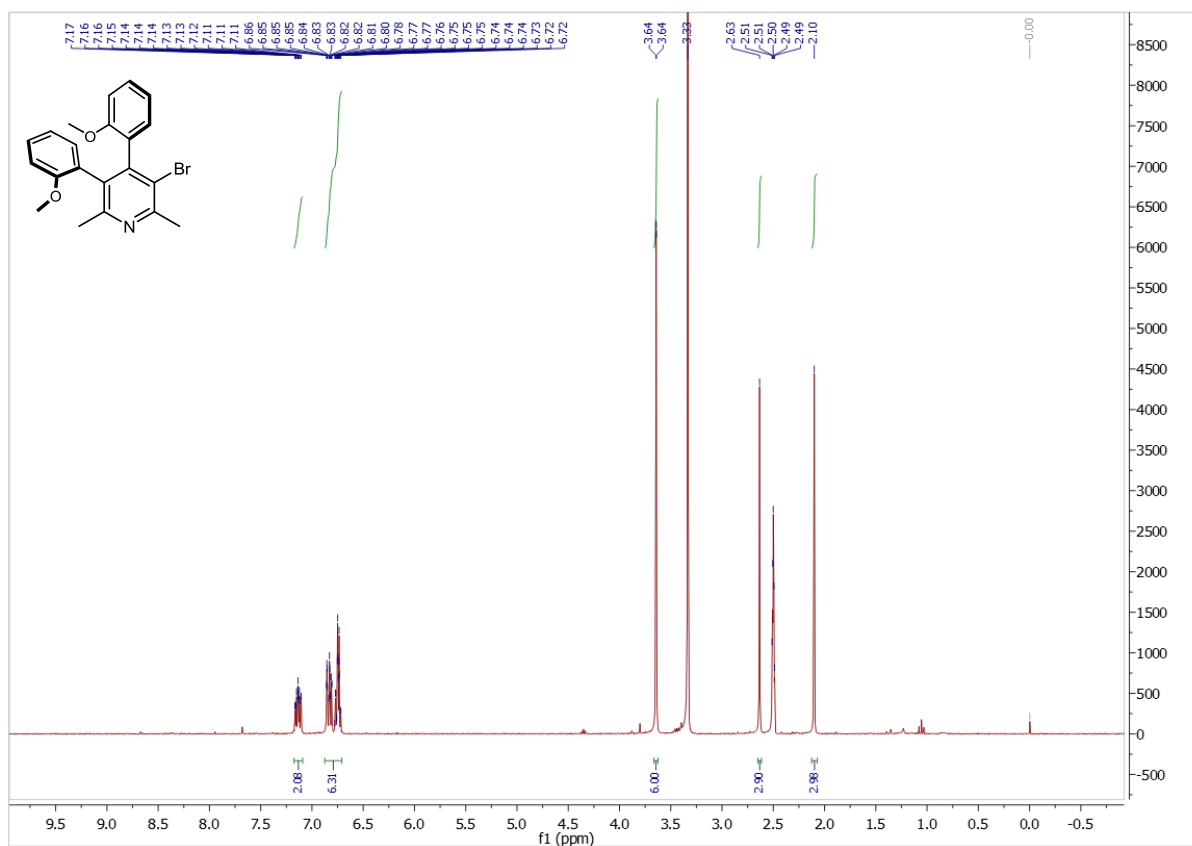
¹³C NMR spectrum of **8** (75 MHz, CDCl₃)



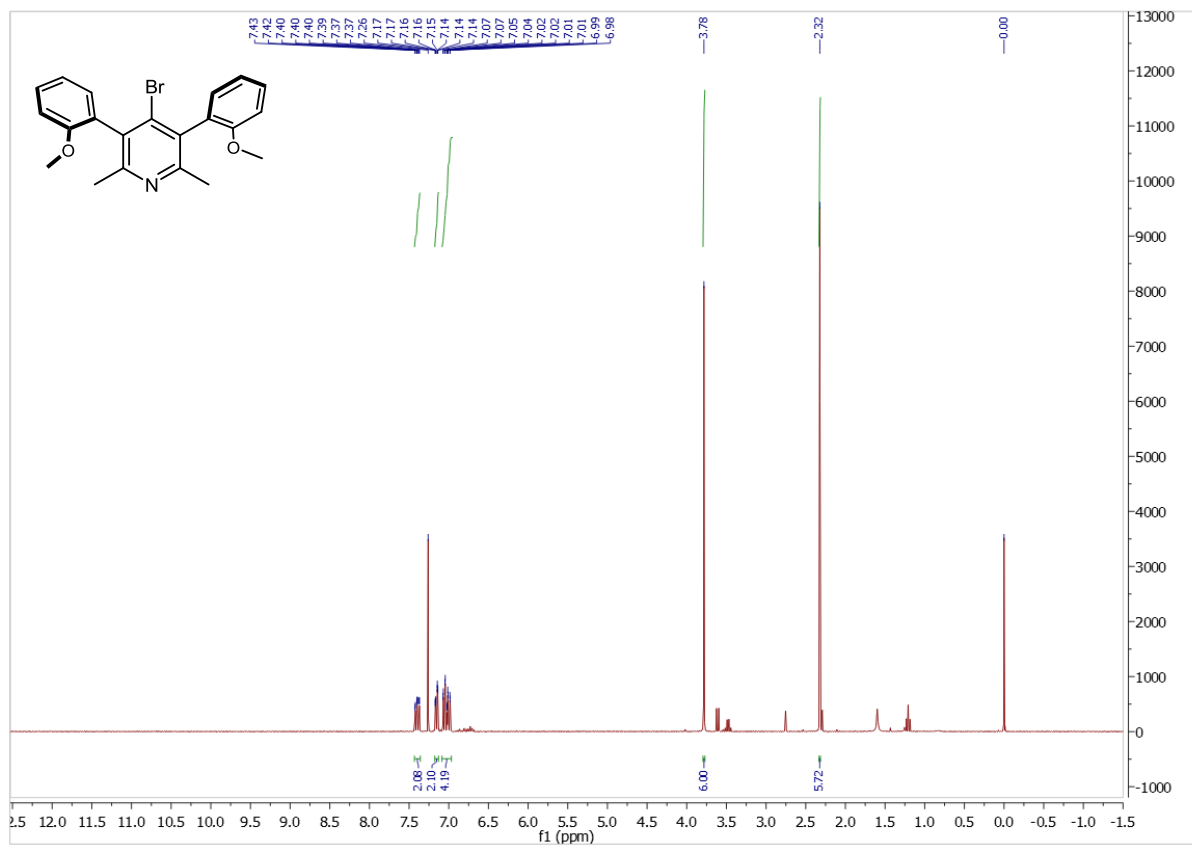
^1H NMR spectrum of **7** (300 MHz, DMSO)



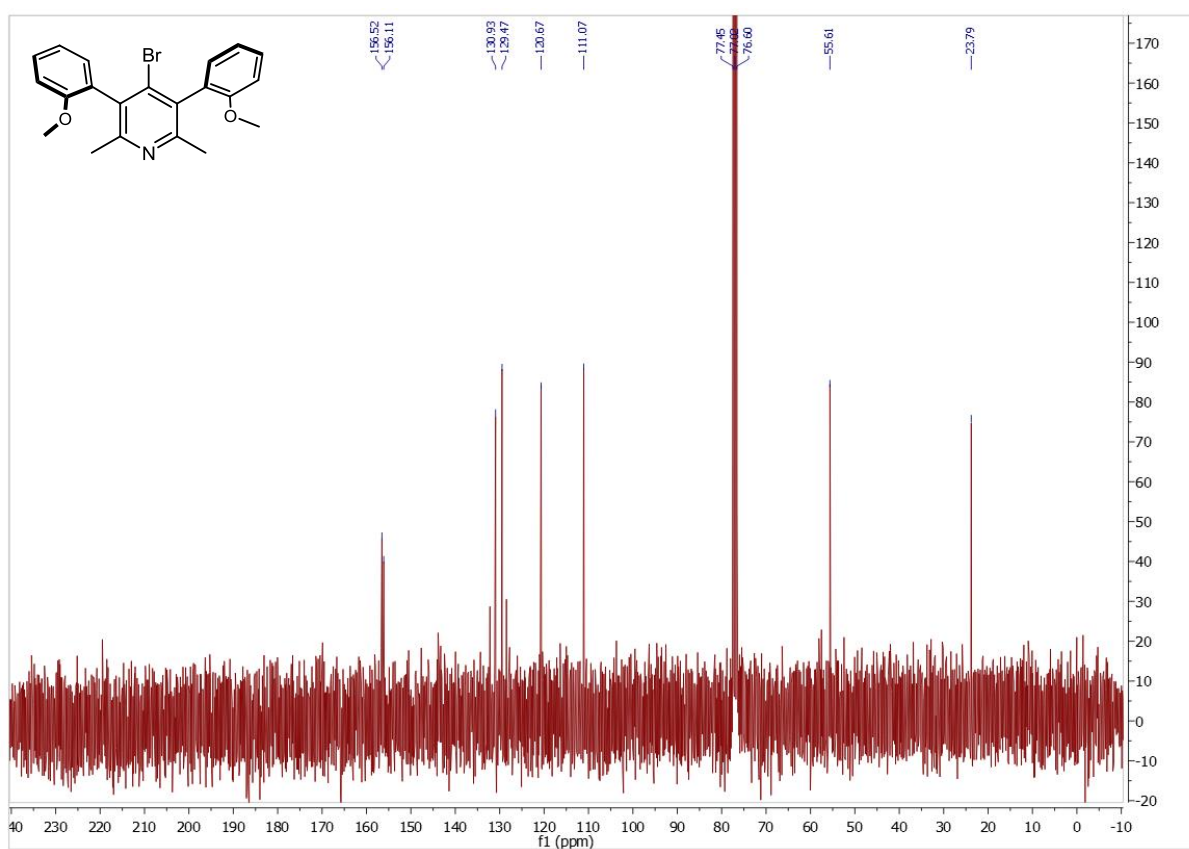
^1H NMR spectrum of **8** (300 MHz, DMSO)



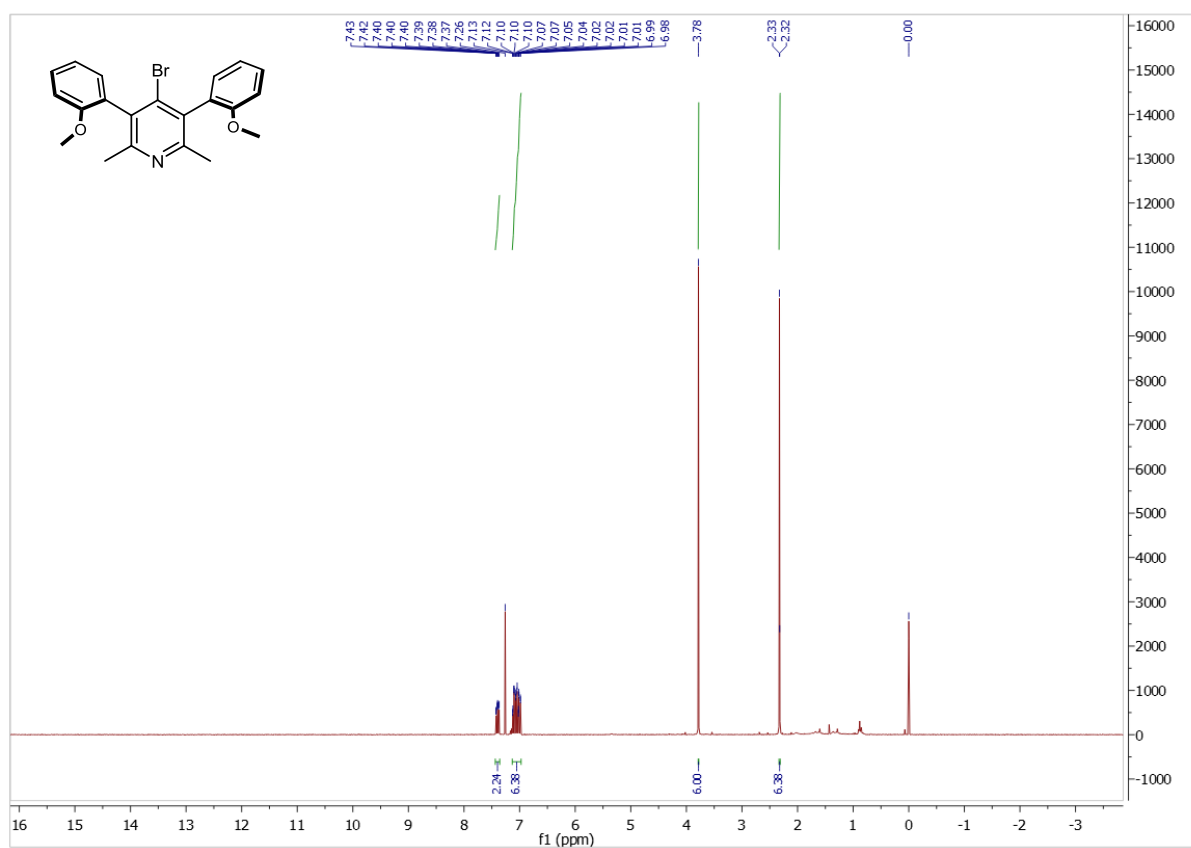
^1H NMR spectrum of **9** (300 MHz, CDCl_3)



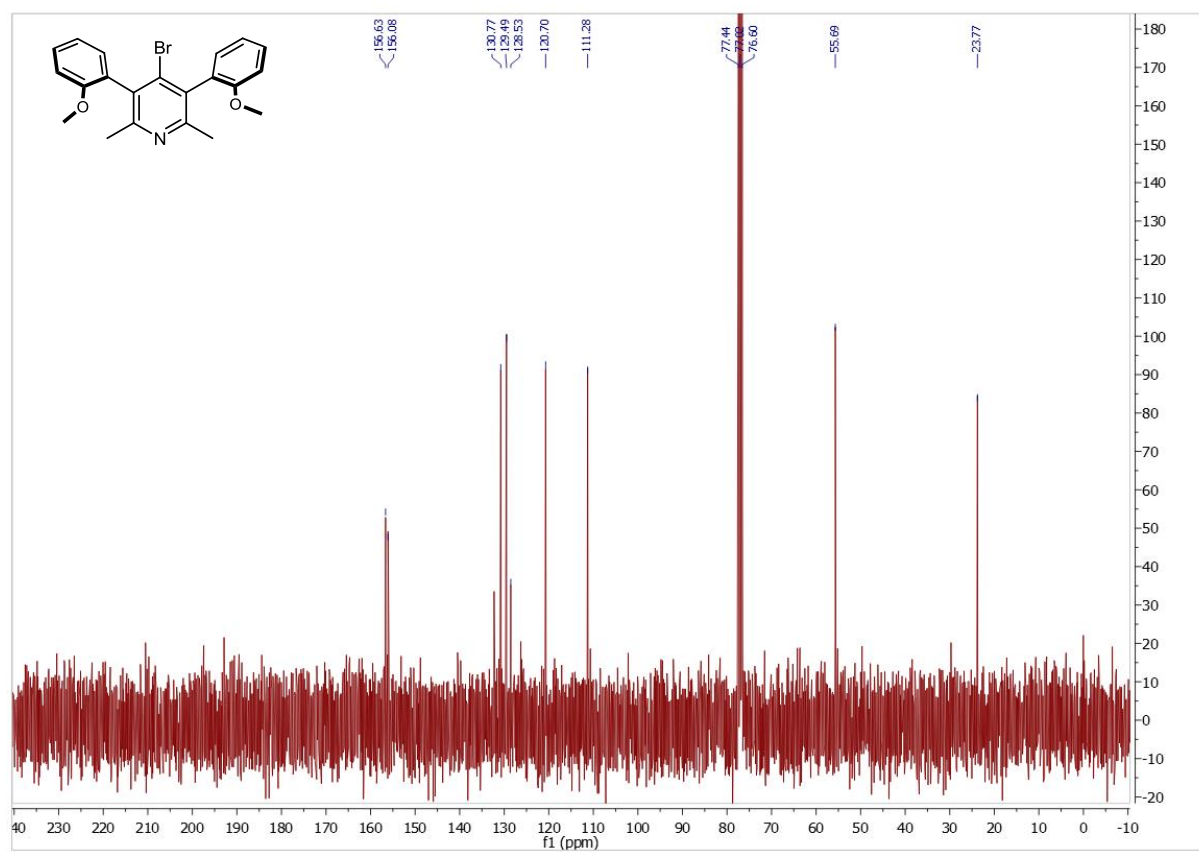
^{13}C NMR spectrum of **9** (75 MHz, CDCl_3)



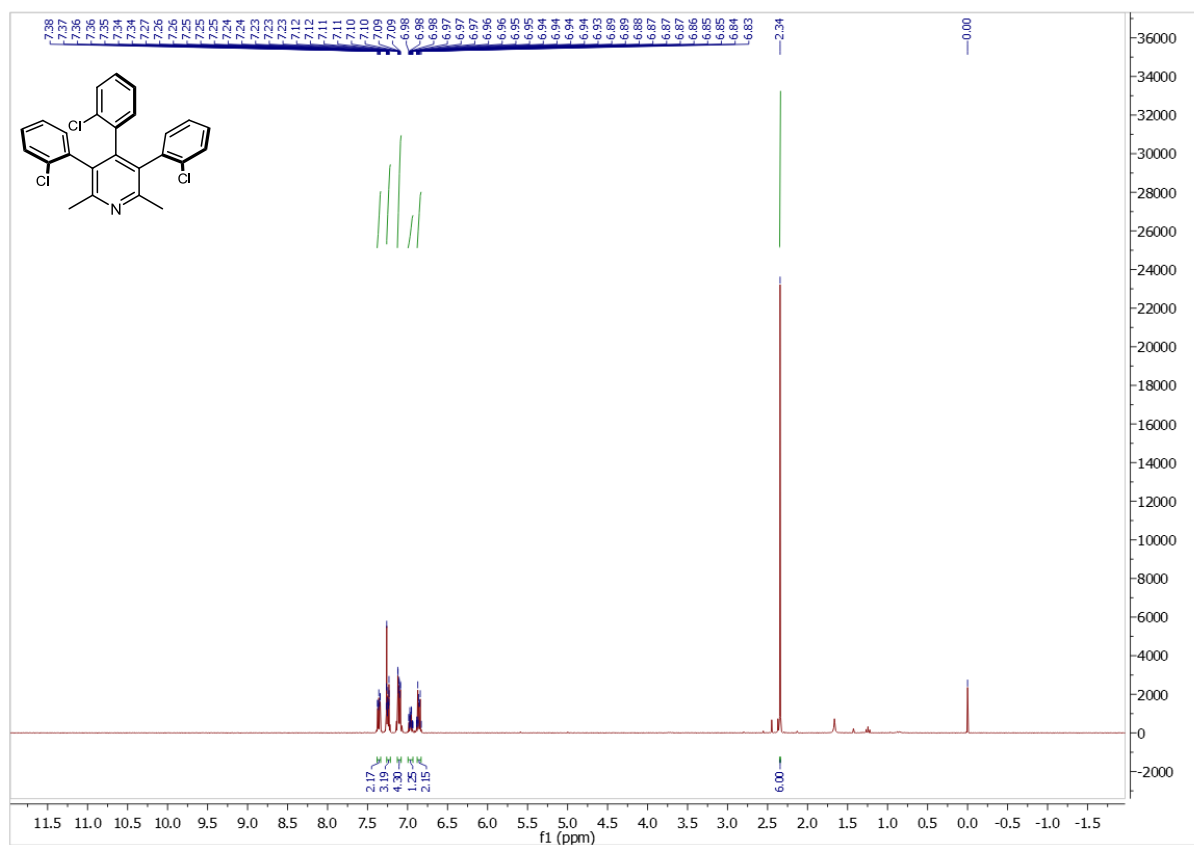
^1H NMR spectrum of **10** (300 MHz, CDCl_3)



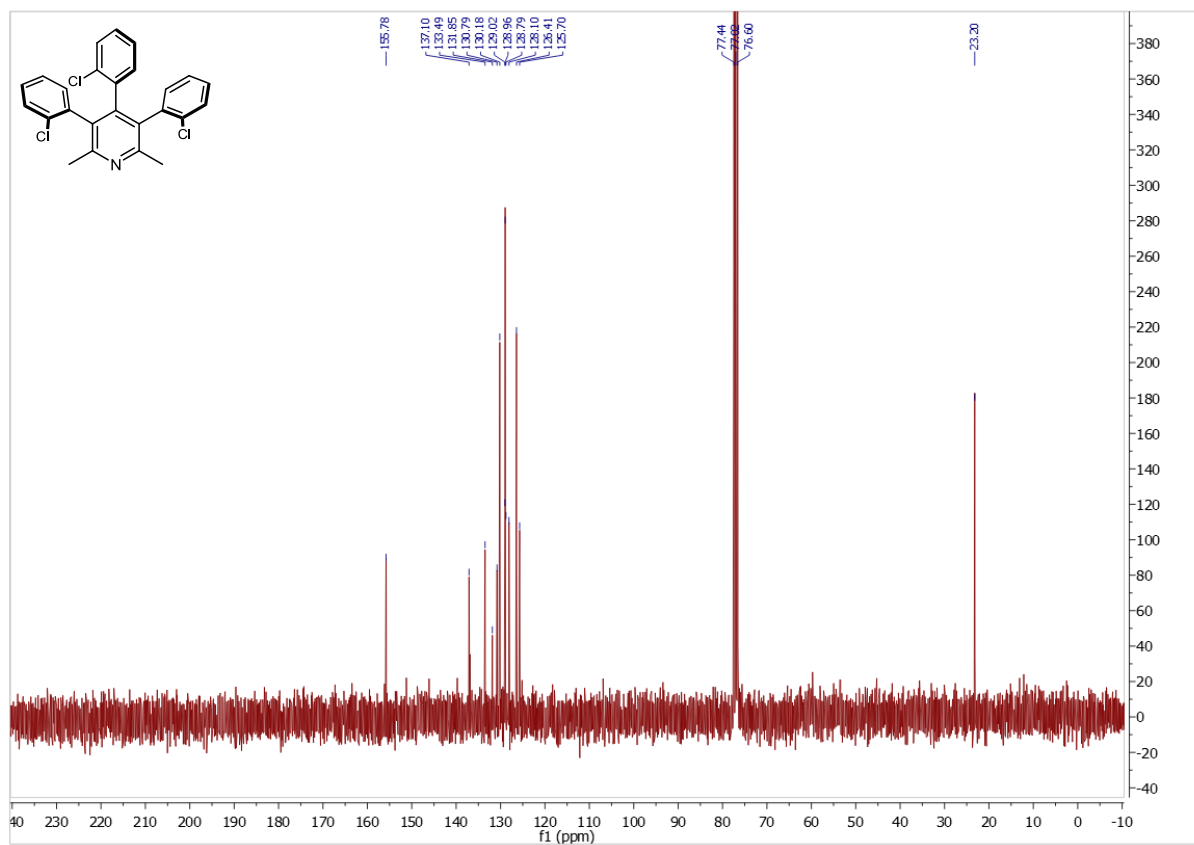
^{13}C NMR spectrum of **10** (75 MHz, CDCl_3)



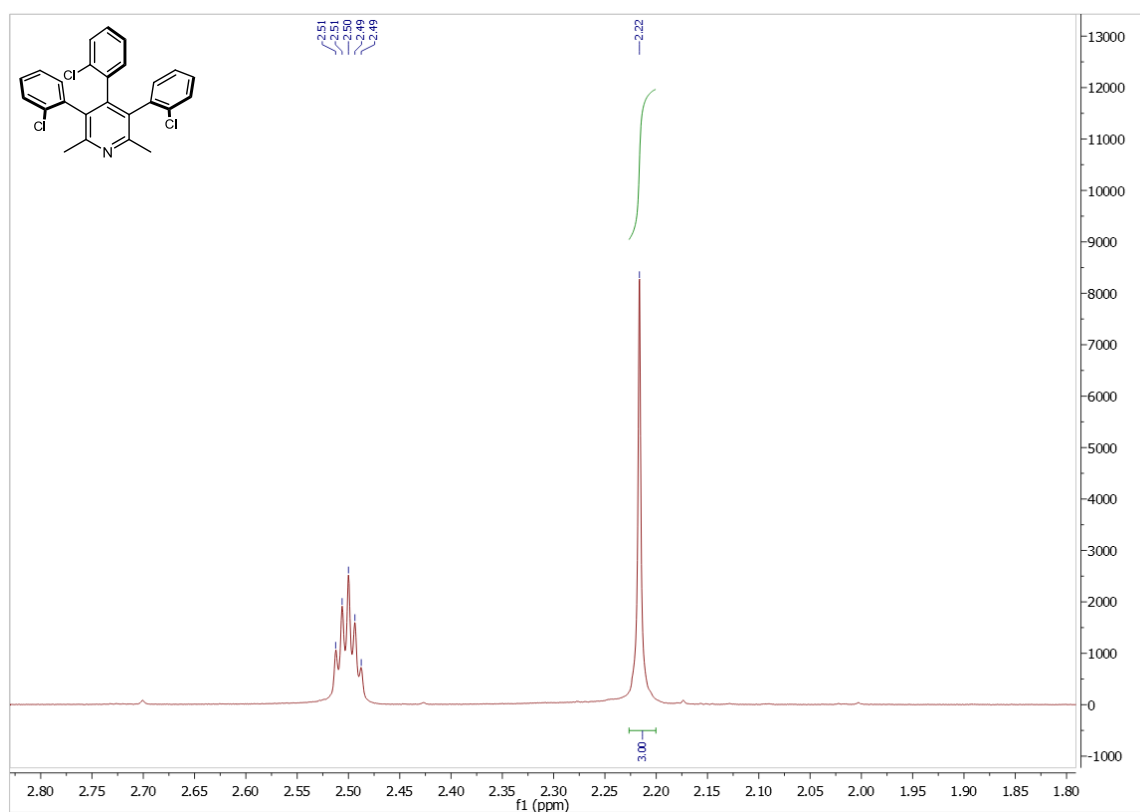
^1H NMR spectrum of **12** (300 MHz, CDCl_3)



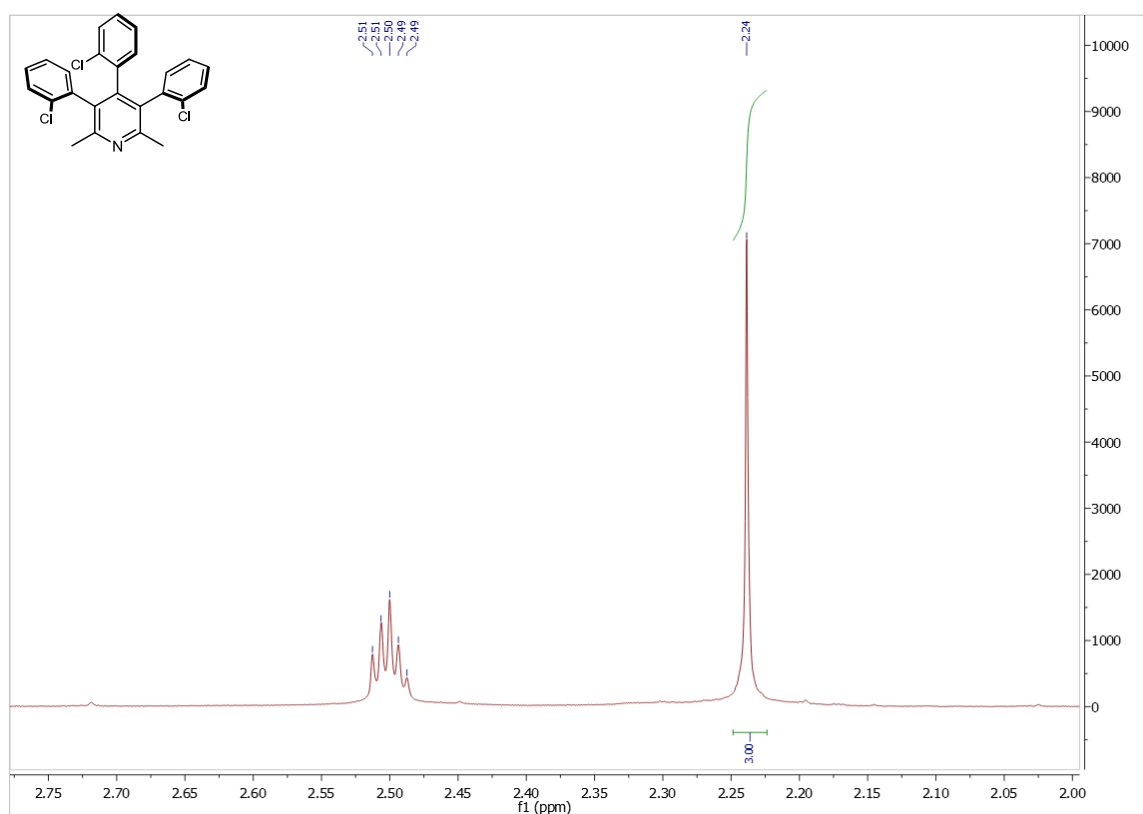
^{13}C NMR spectrum of **12** (75 MHz, CDCl_3)



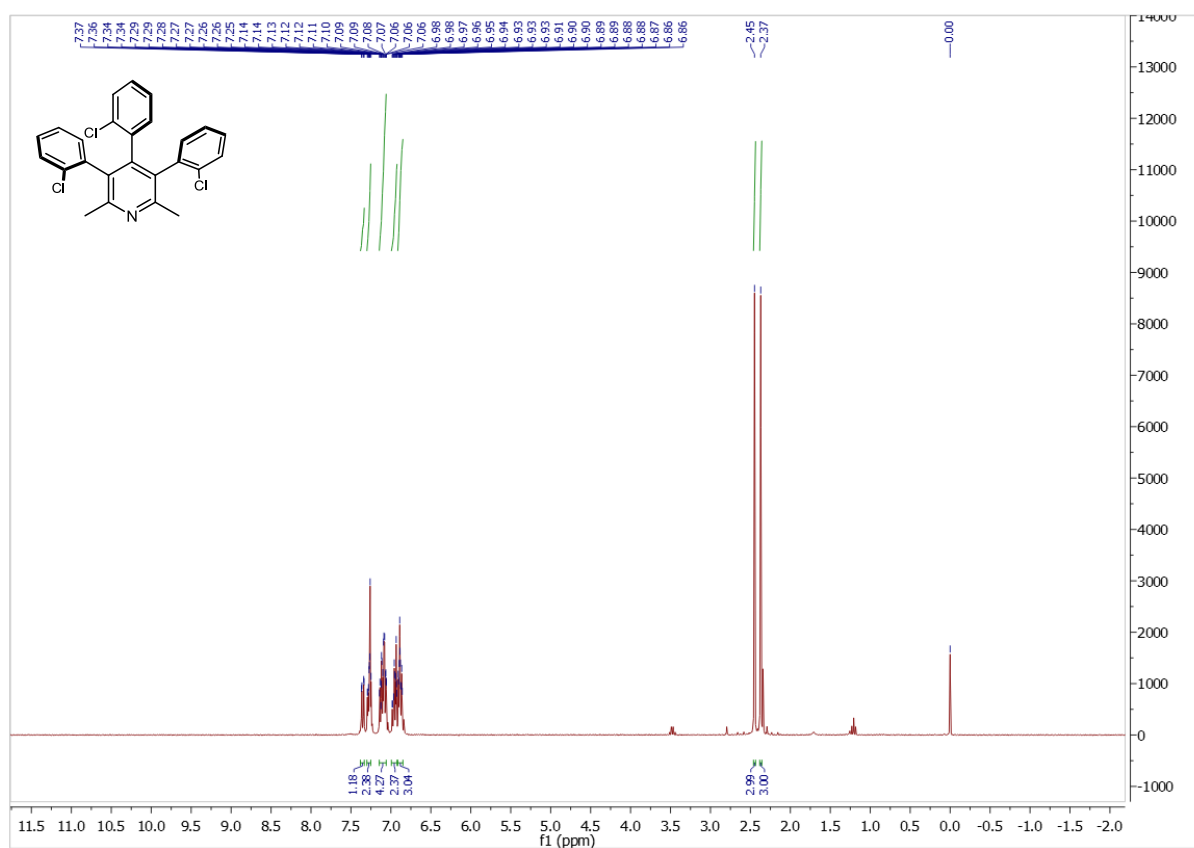
^1H NMR spectrum of **12** (300 MHz, DMSO) $t=25^\circ\text{C}$



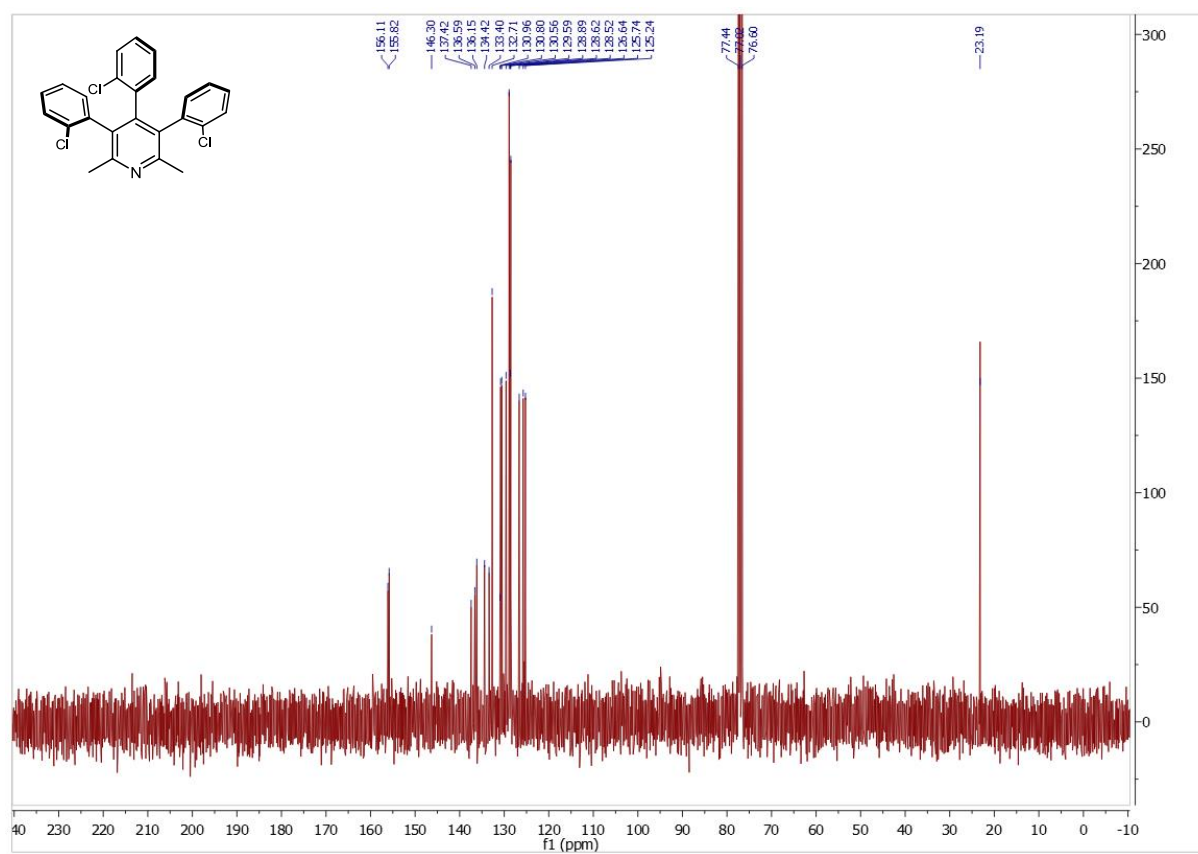
^1H NMR spectrum of **12** (300 MHz, DMSO) after 2 h heating at 120°C ,



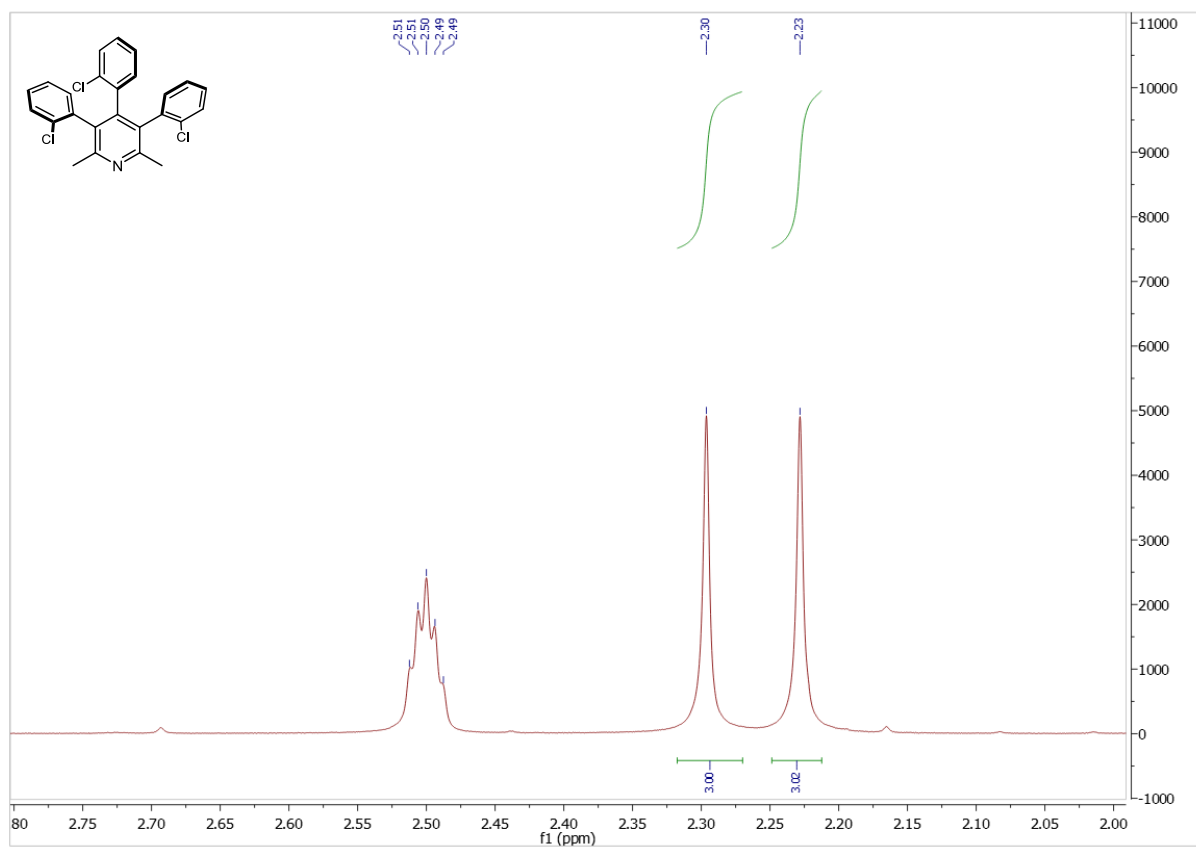
^1H NMR spectrum of **13** (300 MHz, CDCl_3)



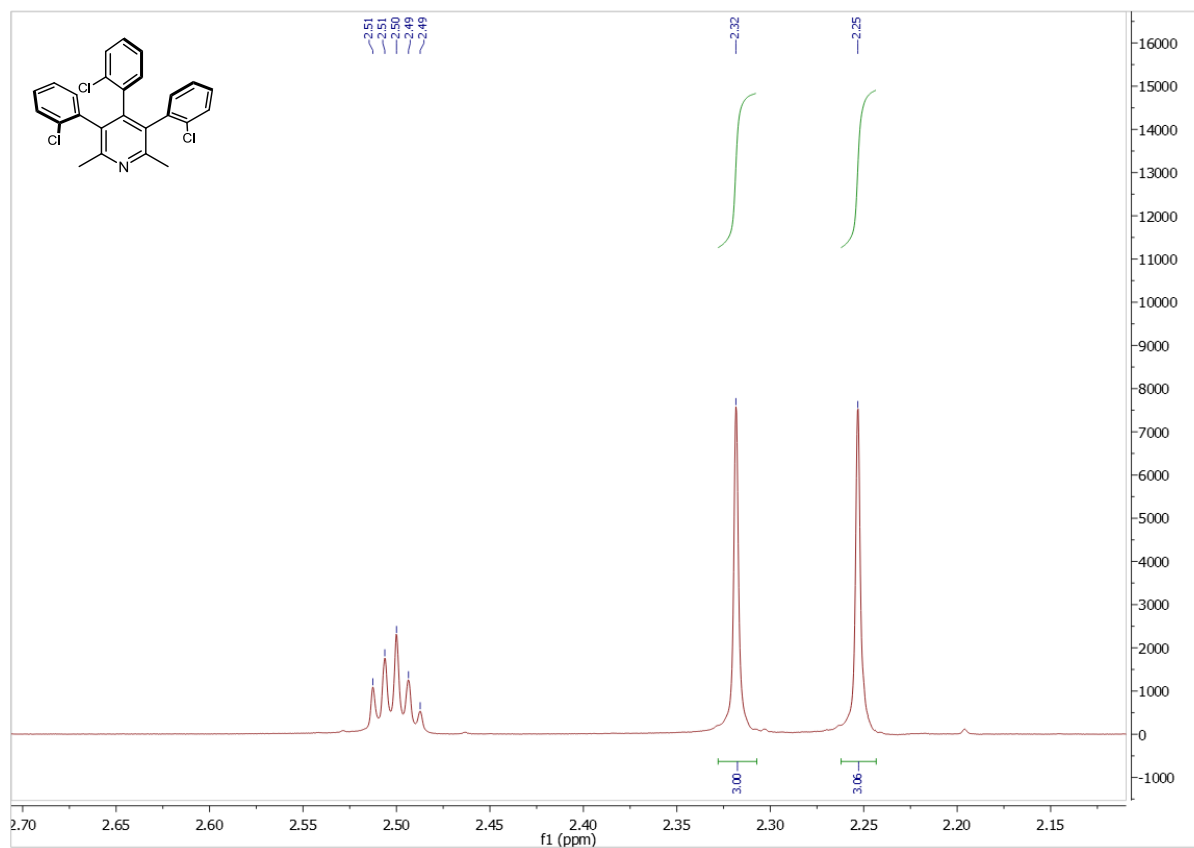
^{13}C NMR spectrum of **13** (75 MHz, CDCl_3)



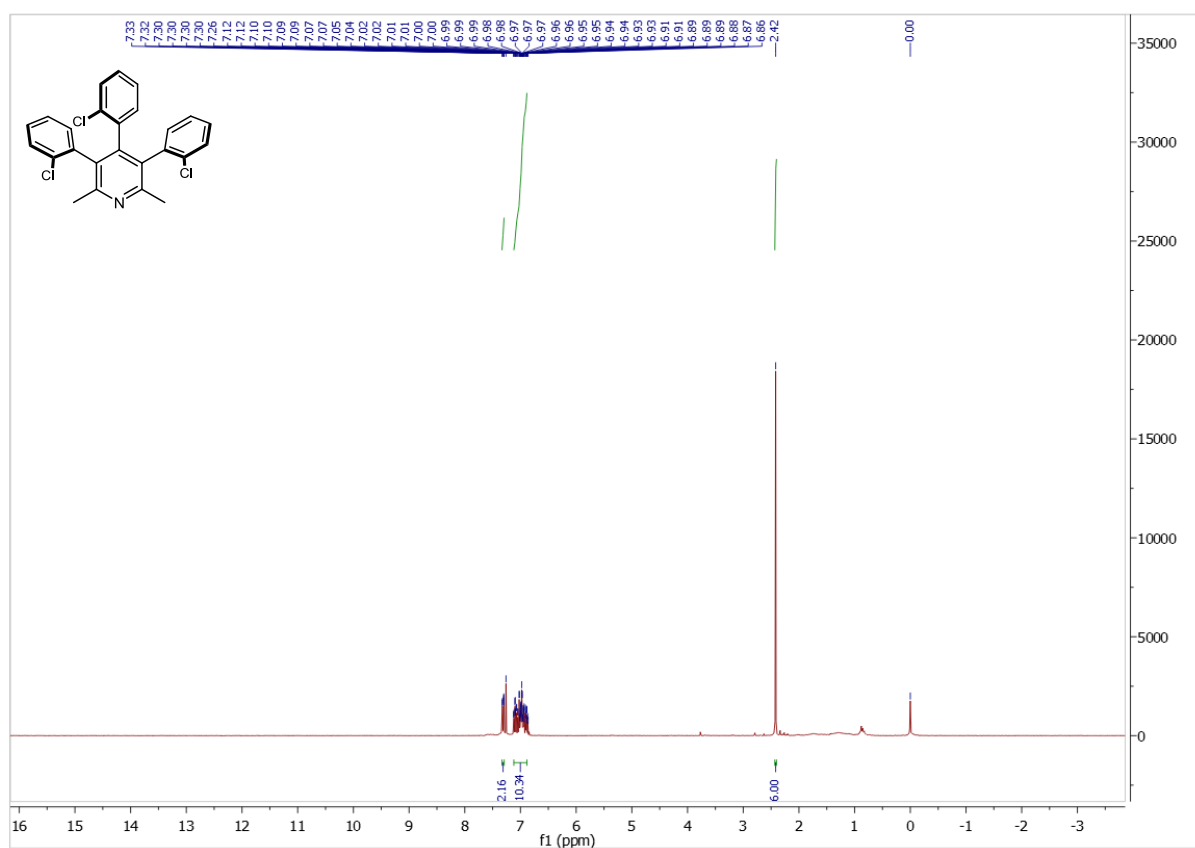
^1H NMR spectrum of **13** (300 MHz, DMSO) $t=25^\circ\text{C}$



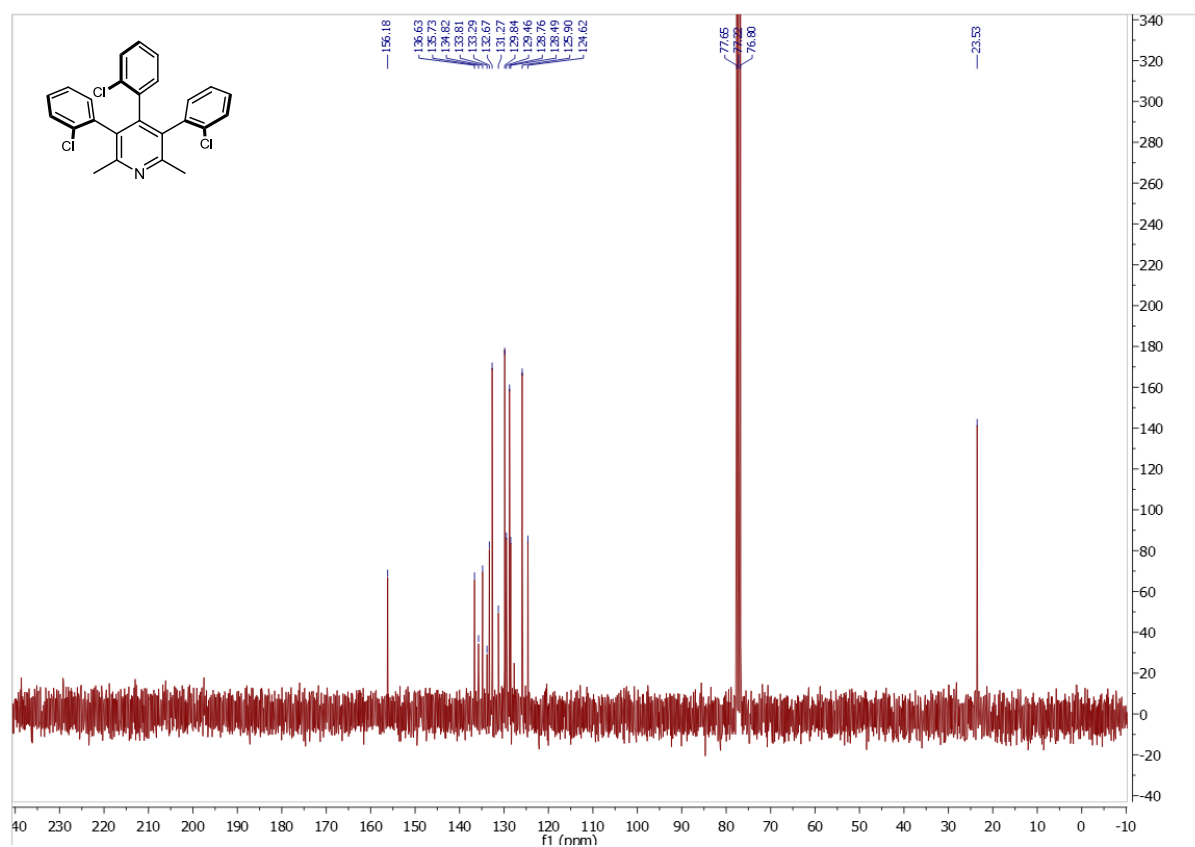
^1H NMR spectrum of **13** (300 MHz, DMSO) after 2 h heating at 120°C



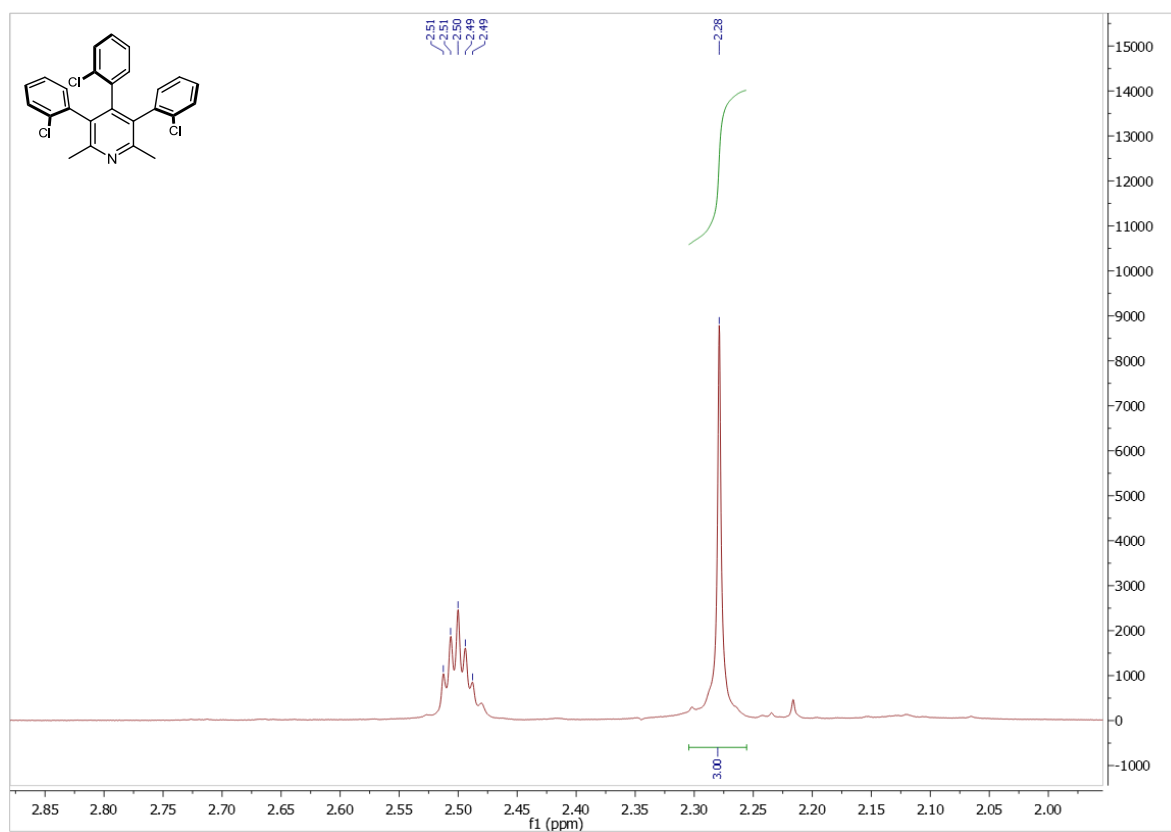
^1H NMR spectrum of **14** (300 MHz, CDCl_3)



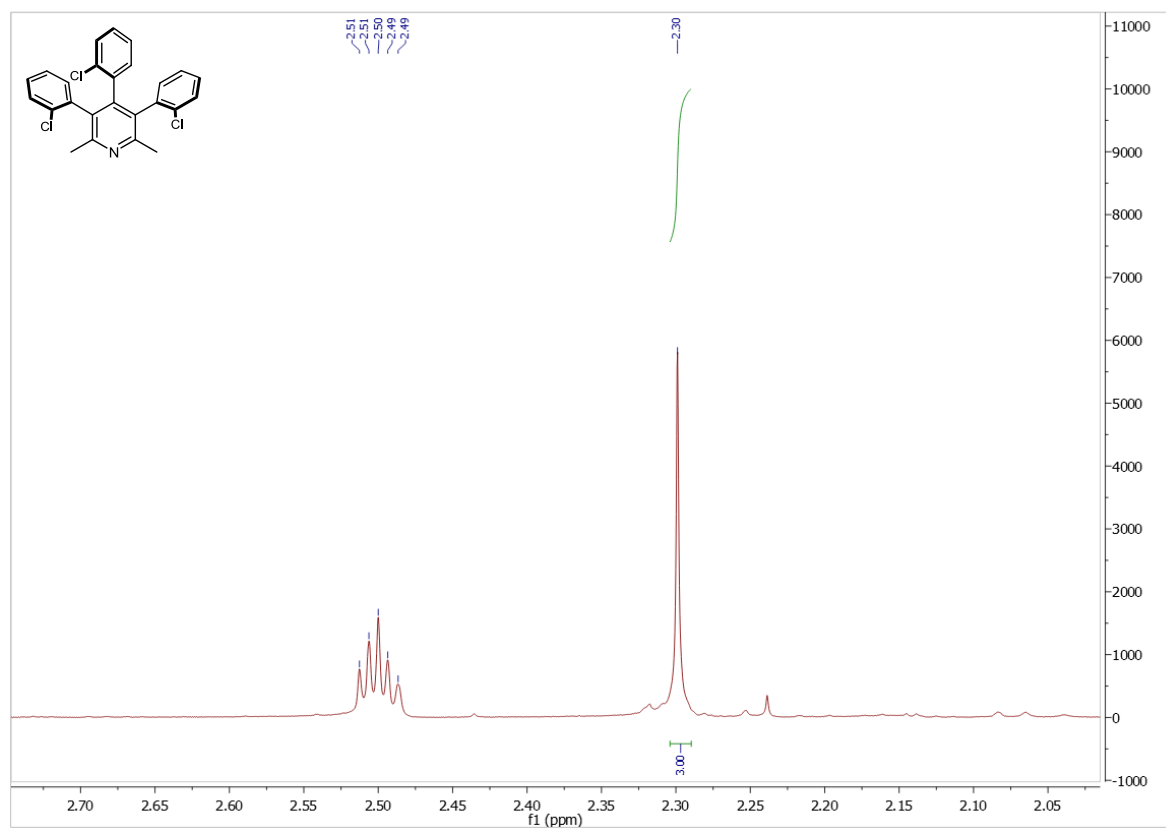
^{13}C NMR spectrum of **14** (75 MHz, CDCl_3)

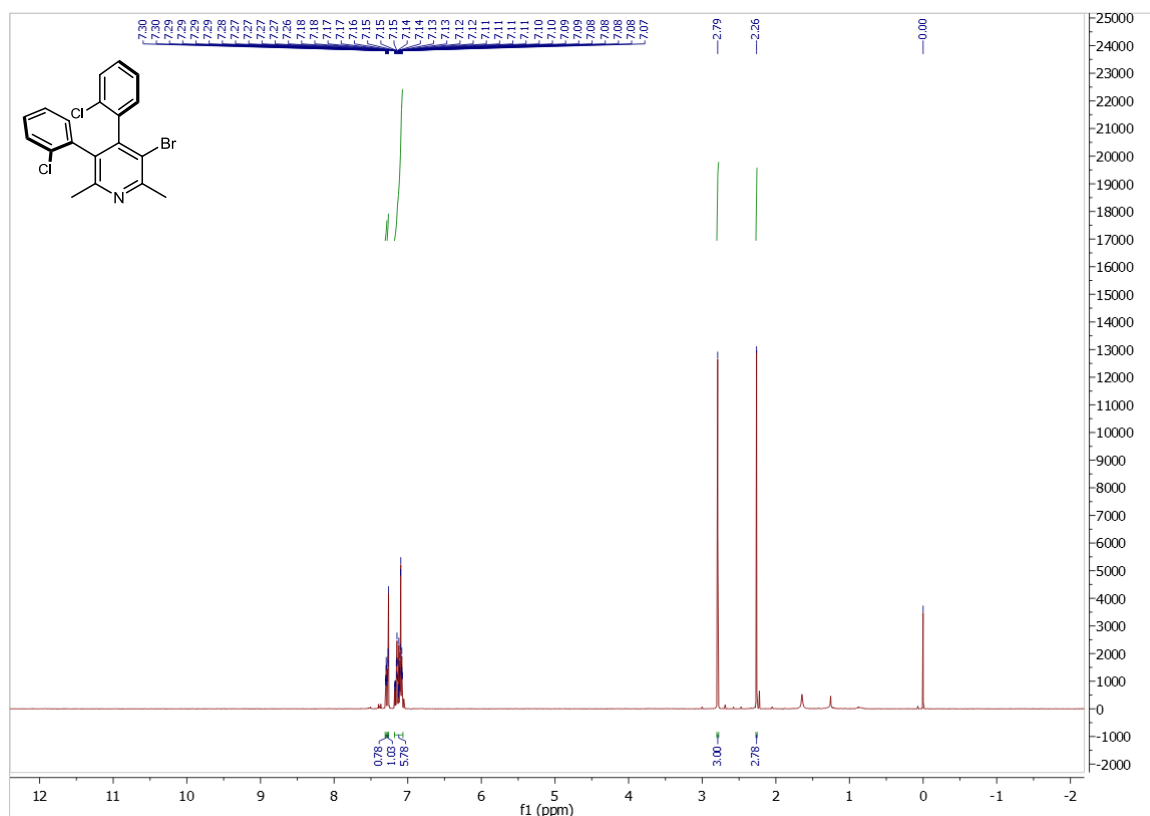


^1H NMR spectrum of **14** (300 MHz, DMSO) $t=25^\circ\text{C}$

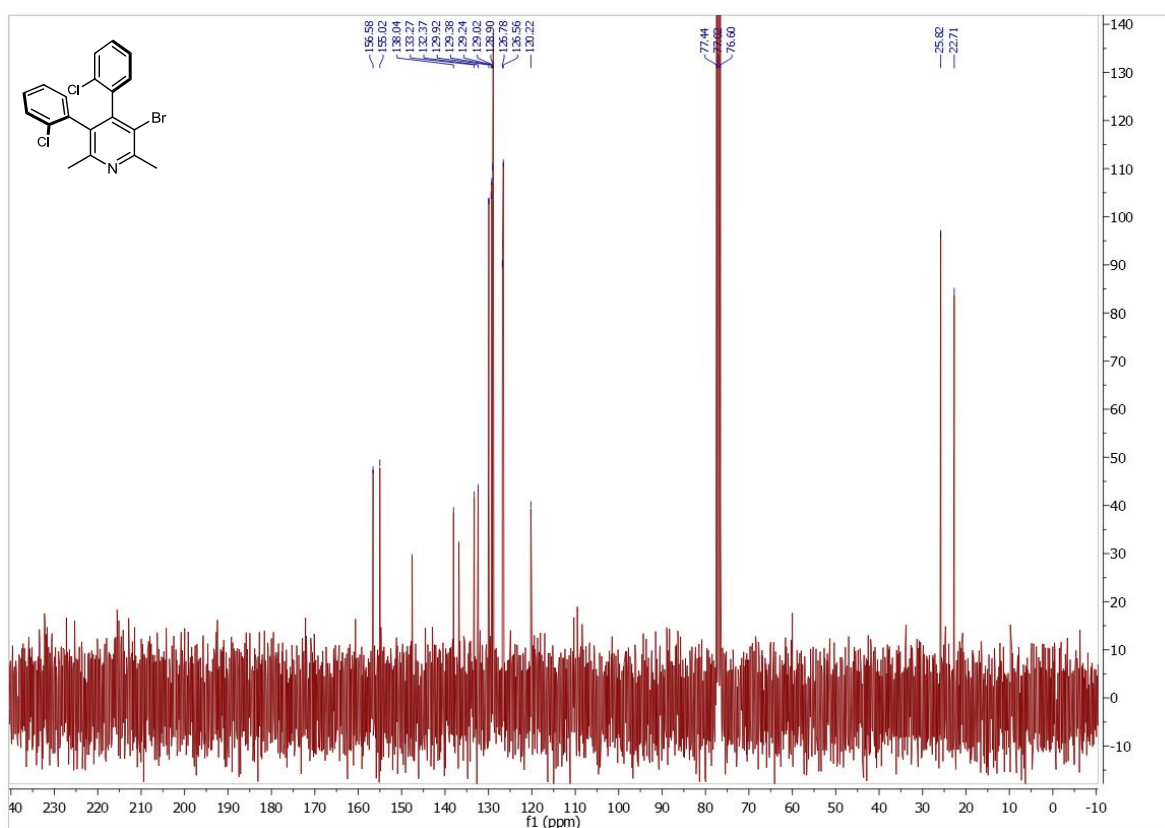


^1H NMR spectrum of **14** (300 MHz, DMSO) after 2 h heating at 120°C

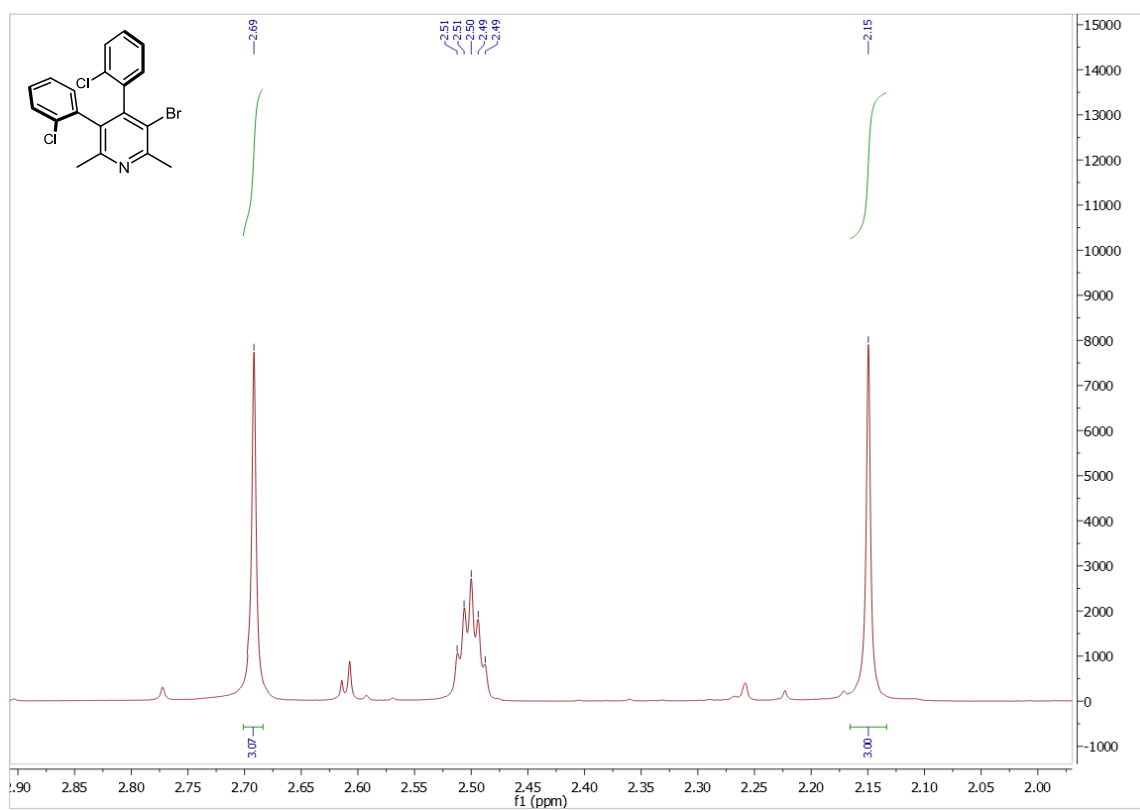


¹H NMR spectrum of **15** (300 MHz, CDCl₃)

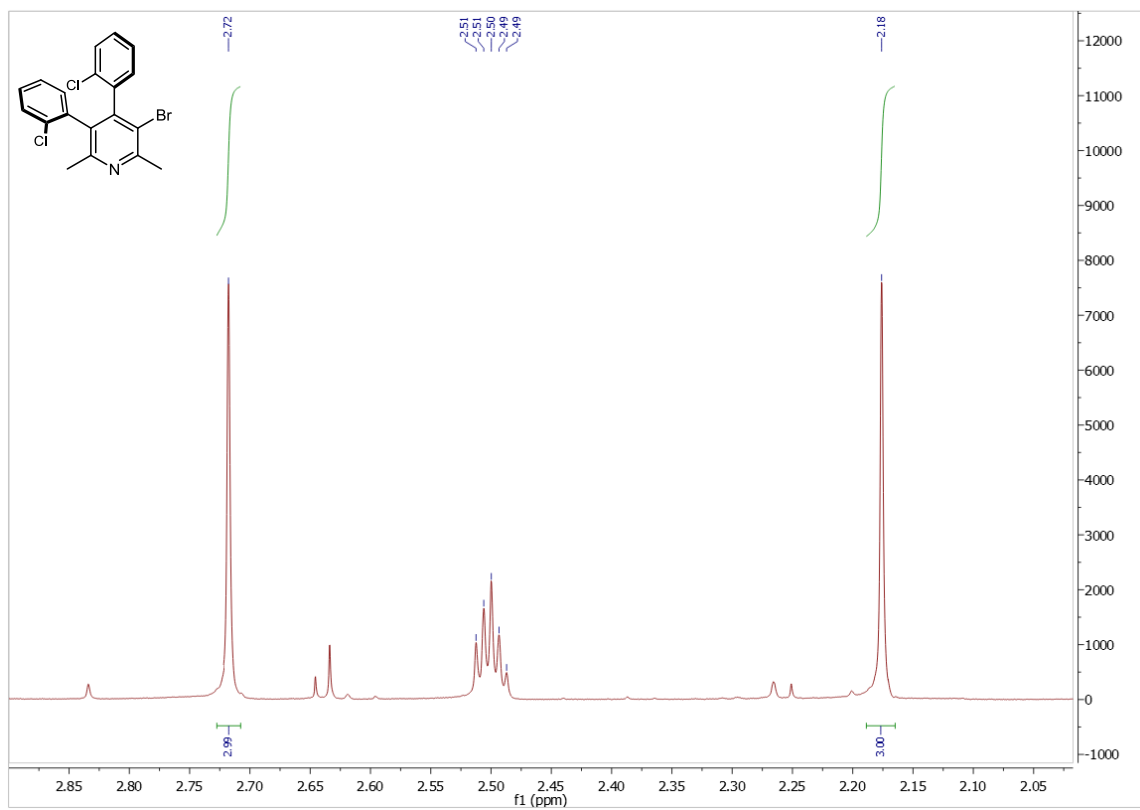
¹³C NMR spectrum of **15** (75 MHz, CDCl₃)



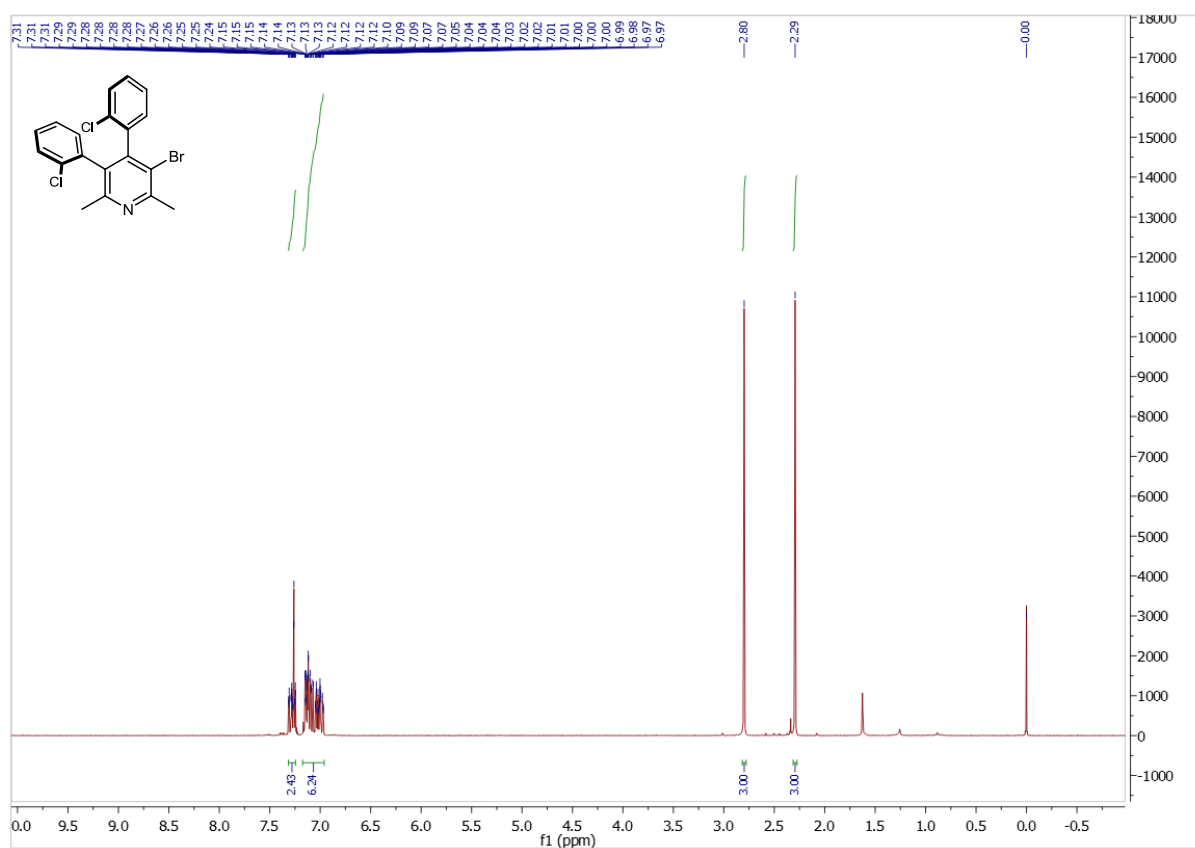
^1H NMR spectrum of **15** (300 MHz, DMSO) $t=25^\circ\text{C}$



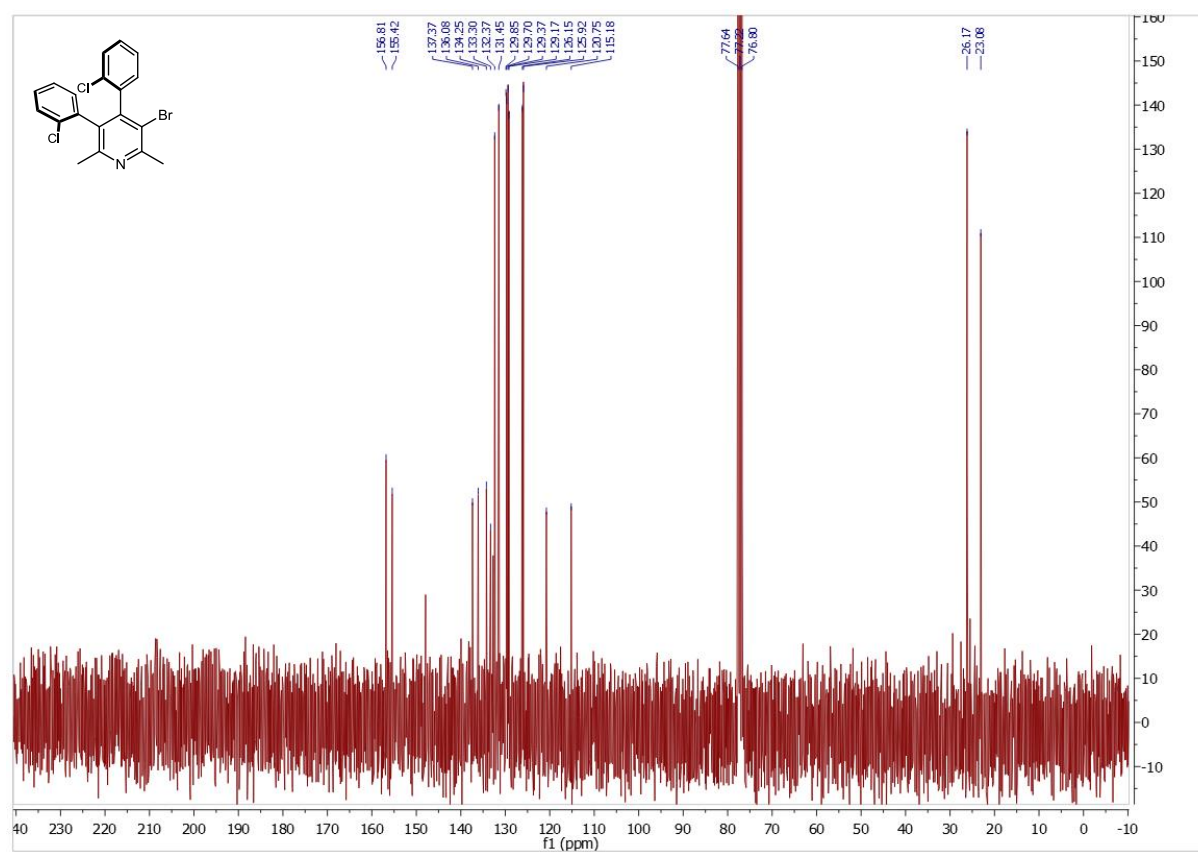
^1H NMR spectrum of **15** (300 MHz, DMSO) after 2 h heating at 120°C



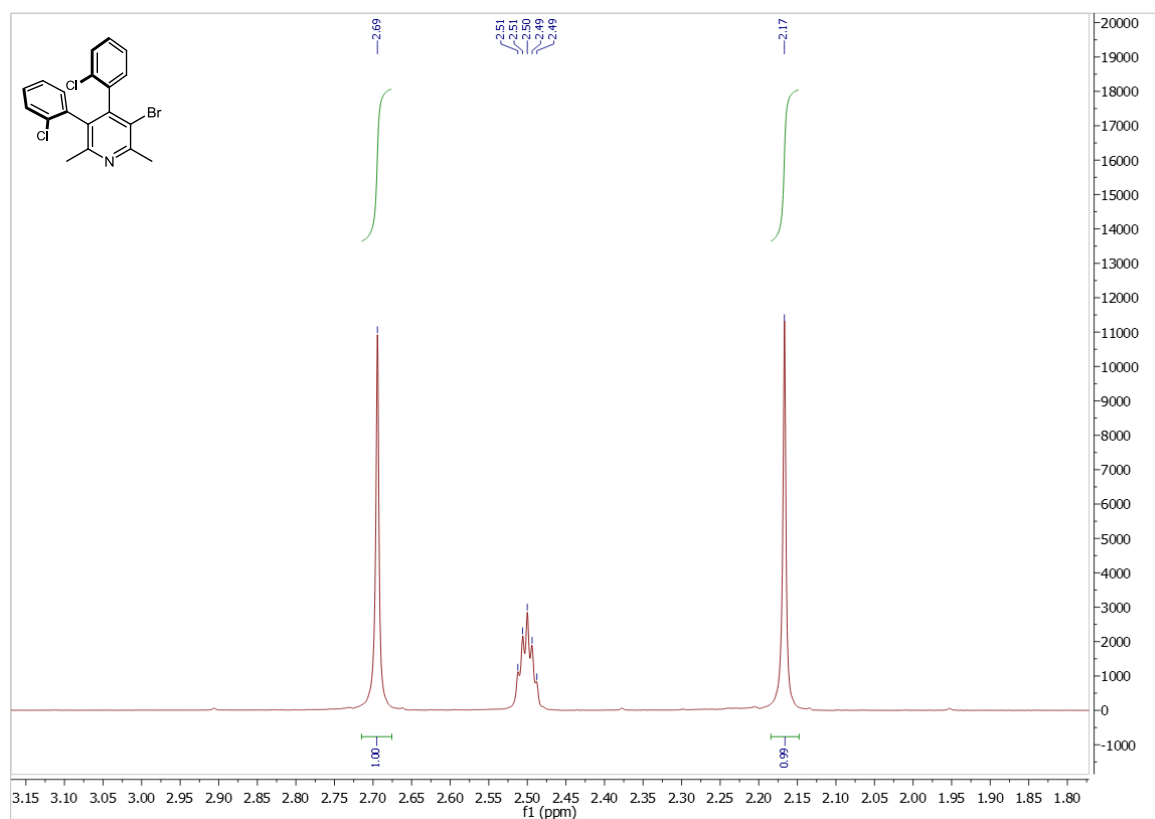
^1H NMR spectrum of **16** (300 MHz, CDCl_3)



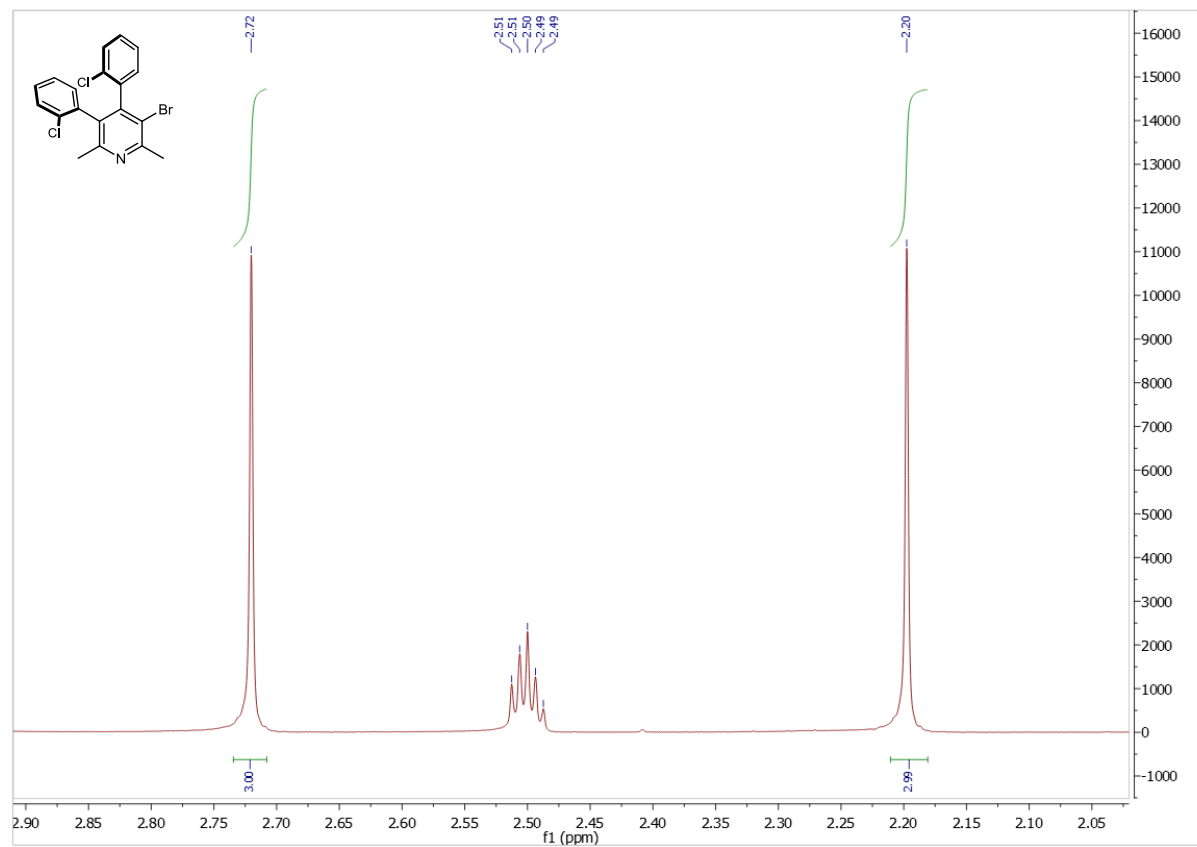
^{13}C NMR spectrum of **16** (75 MHz, CDCl_3)



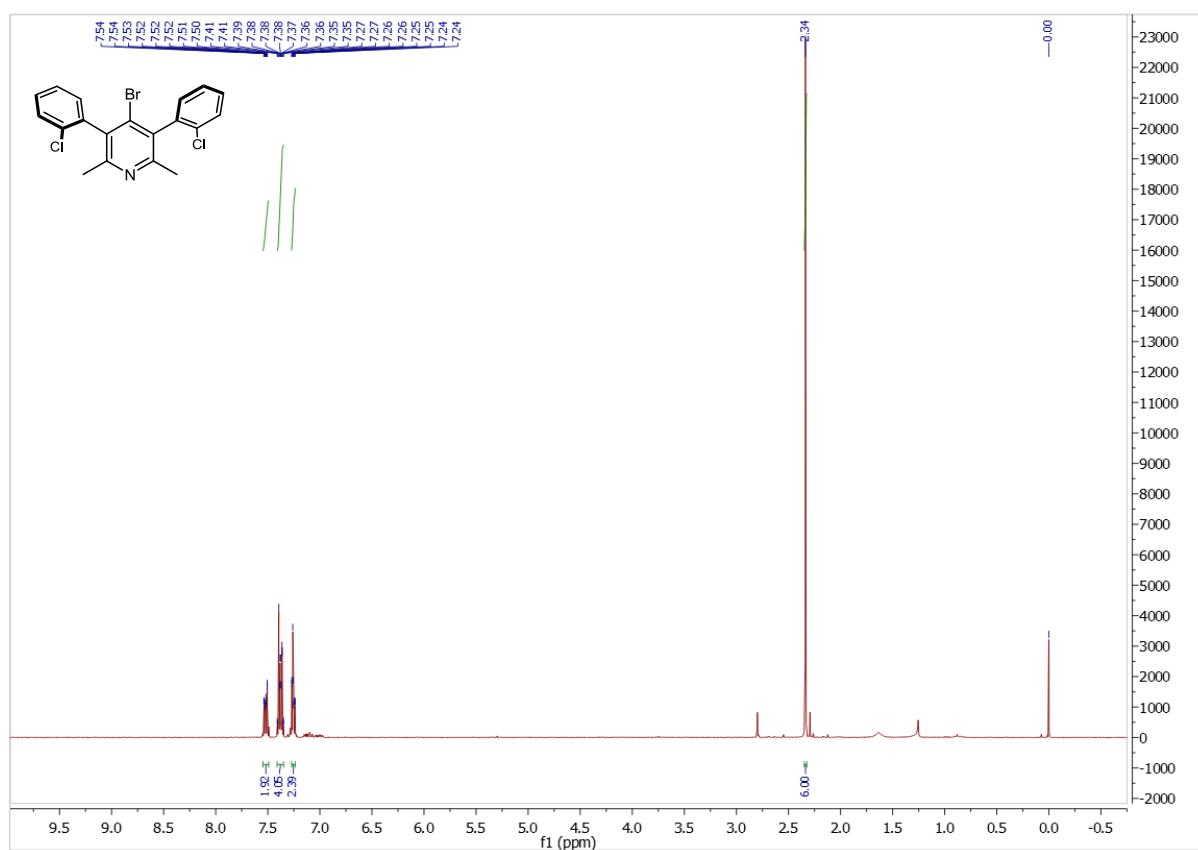
^1H NMR spectrum of **16** (300 MHz, DMSO) $t=25^\circ\text{C}$



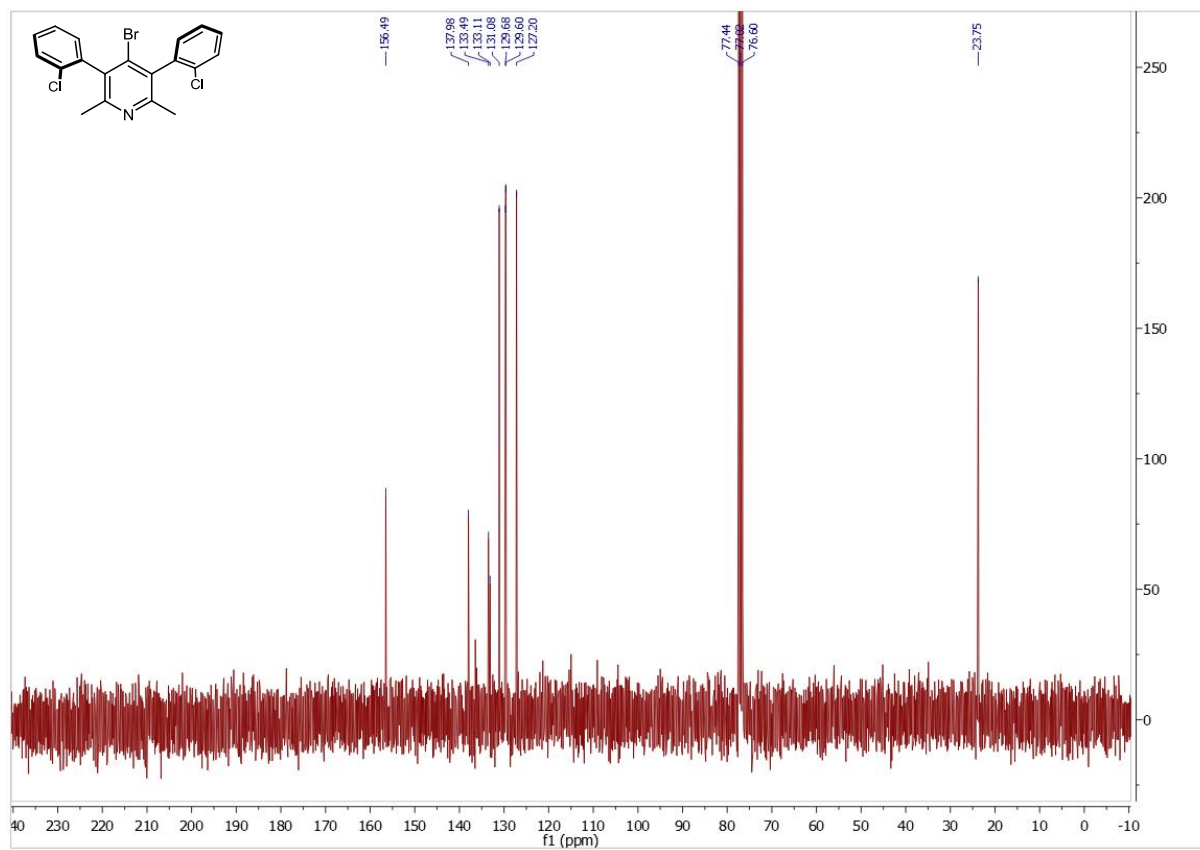
^1H NMR spectrum of **16** (300 MHz, DMSO) after 2 h heating at 120°C



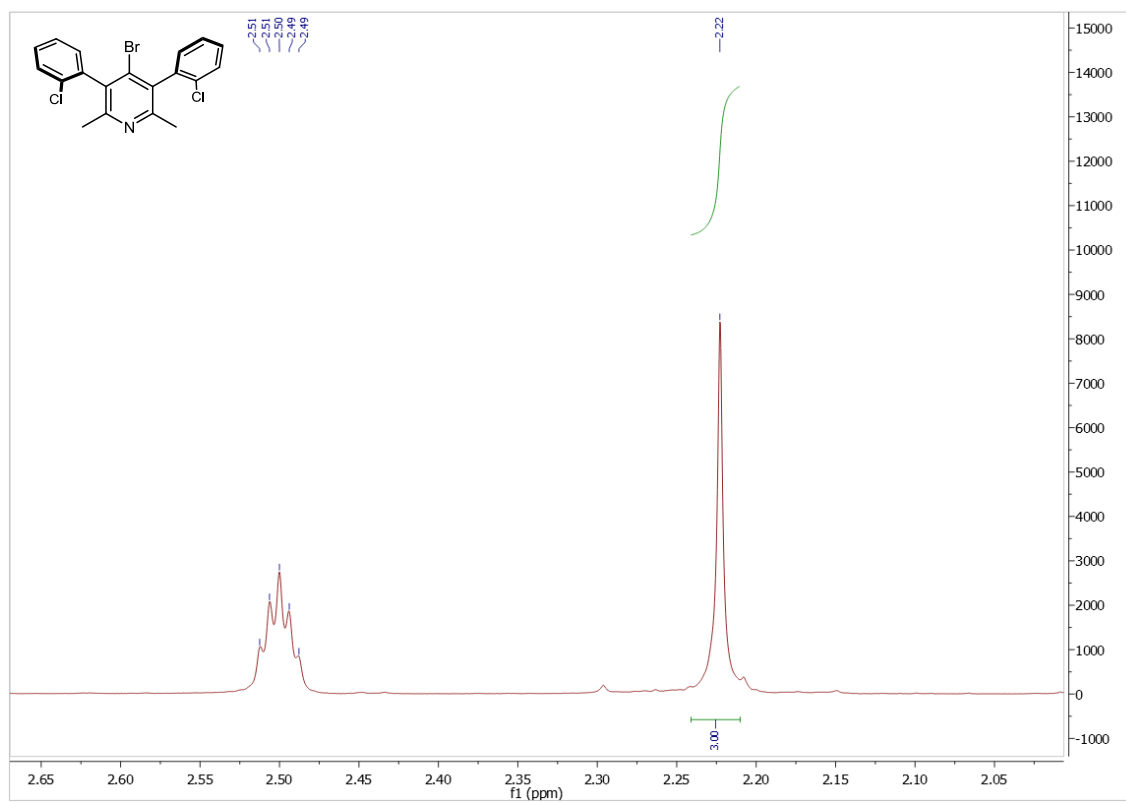
^1H NMR spectrum of **17** (300 MHz, CDCl_3)



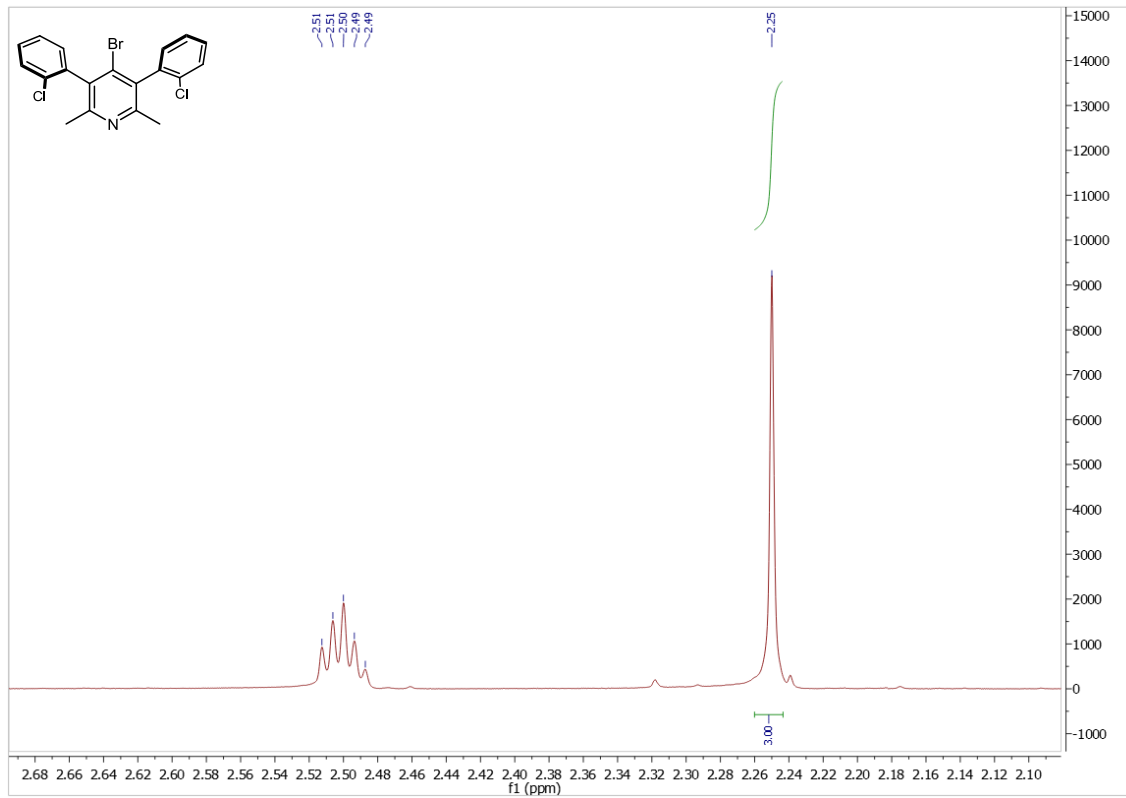
^{13}C NMR spectrum of **17** (75 MHz, CDCl_3)

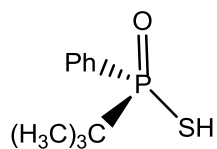


^1H NMR spectrum of **17** (300 MHz, DMSO) $t=25^\circ\text{C}$



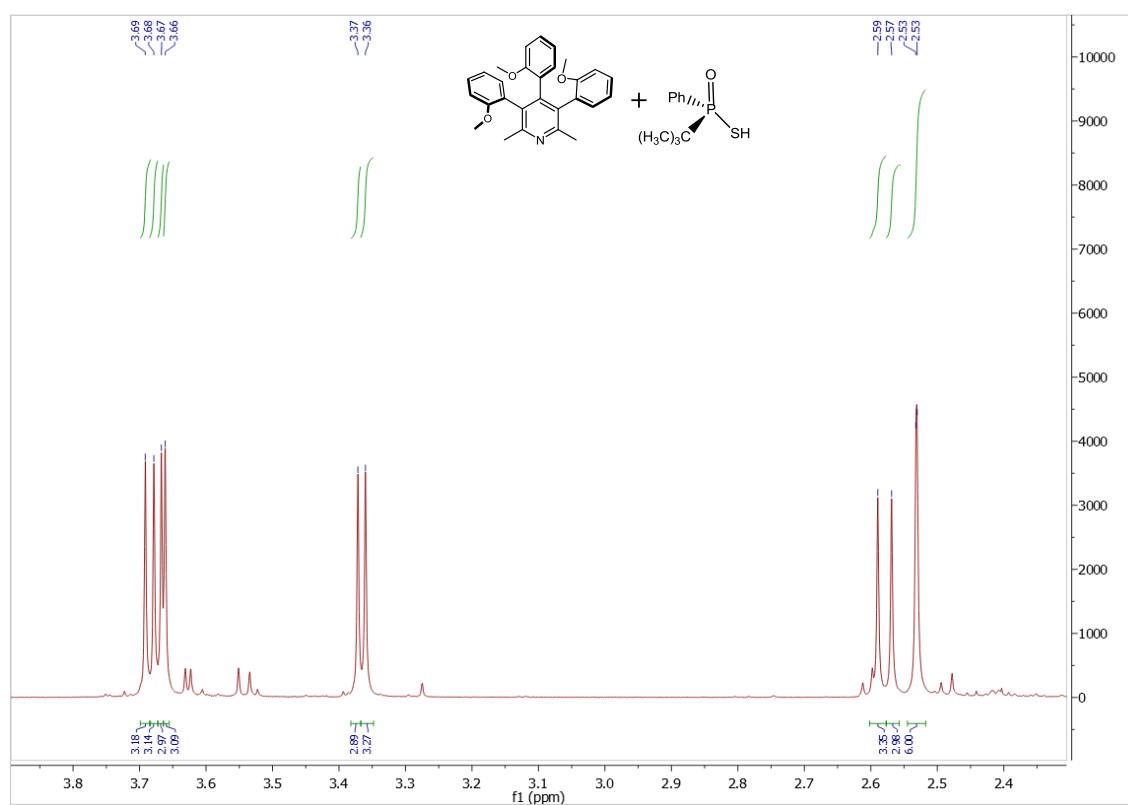
^1H NMR spectrum of **17** (300 MHz, DMSO) after 2 h heating at 120°C



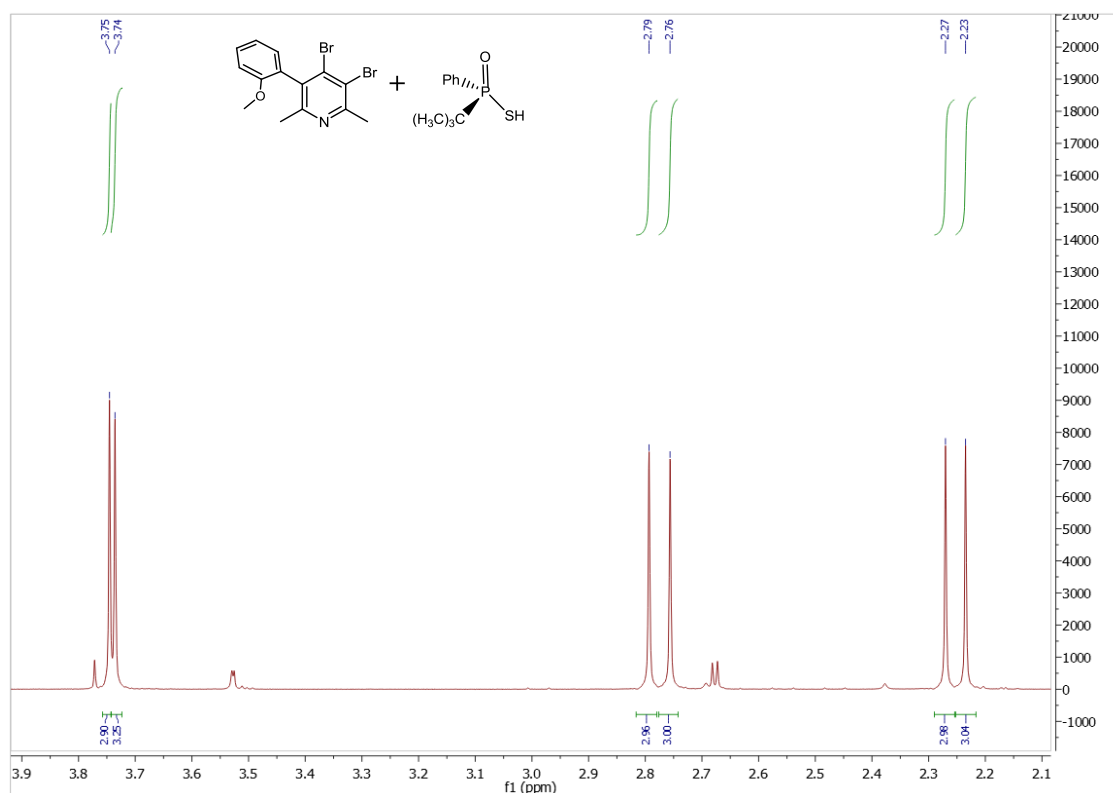


Structure of (+)-(R)-tert-butyl(phenyl) phosphinothioic acid

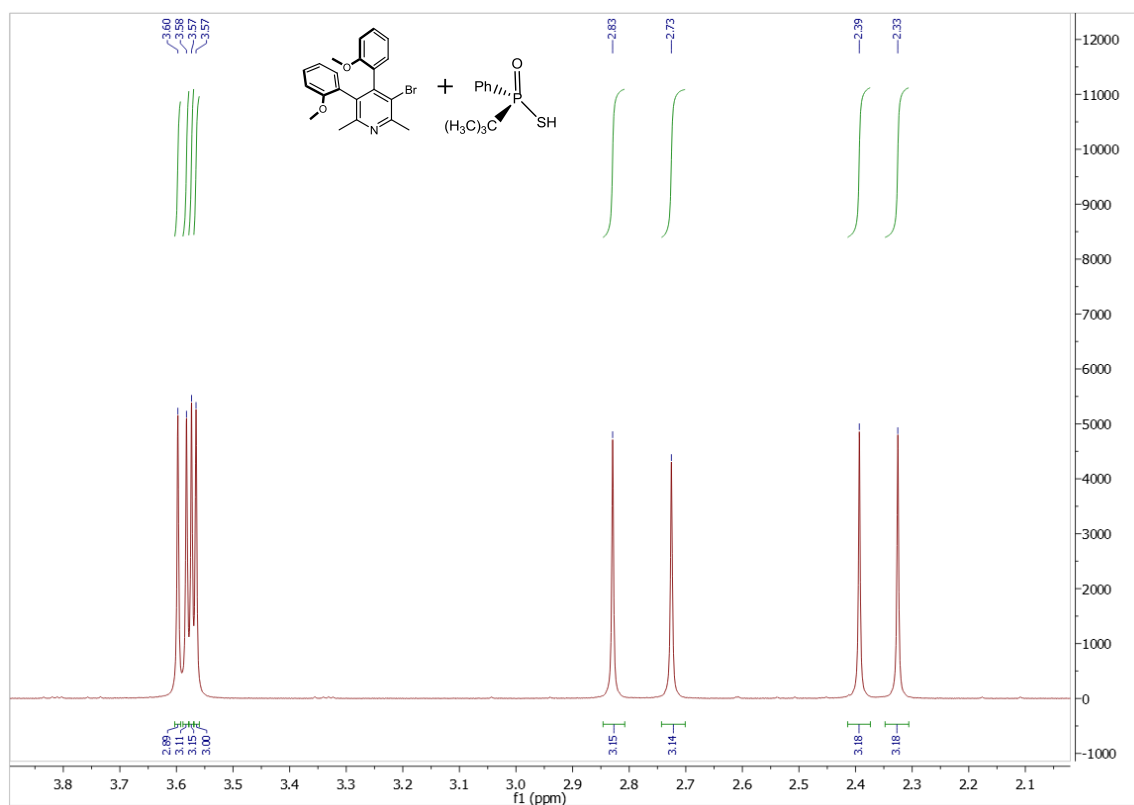
^1H NMR (CH_3 and OCH_3 protons) of racemic **2** in presence of (+)-(R)-tert-butyl(phenyl) phosphinothioic acid



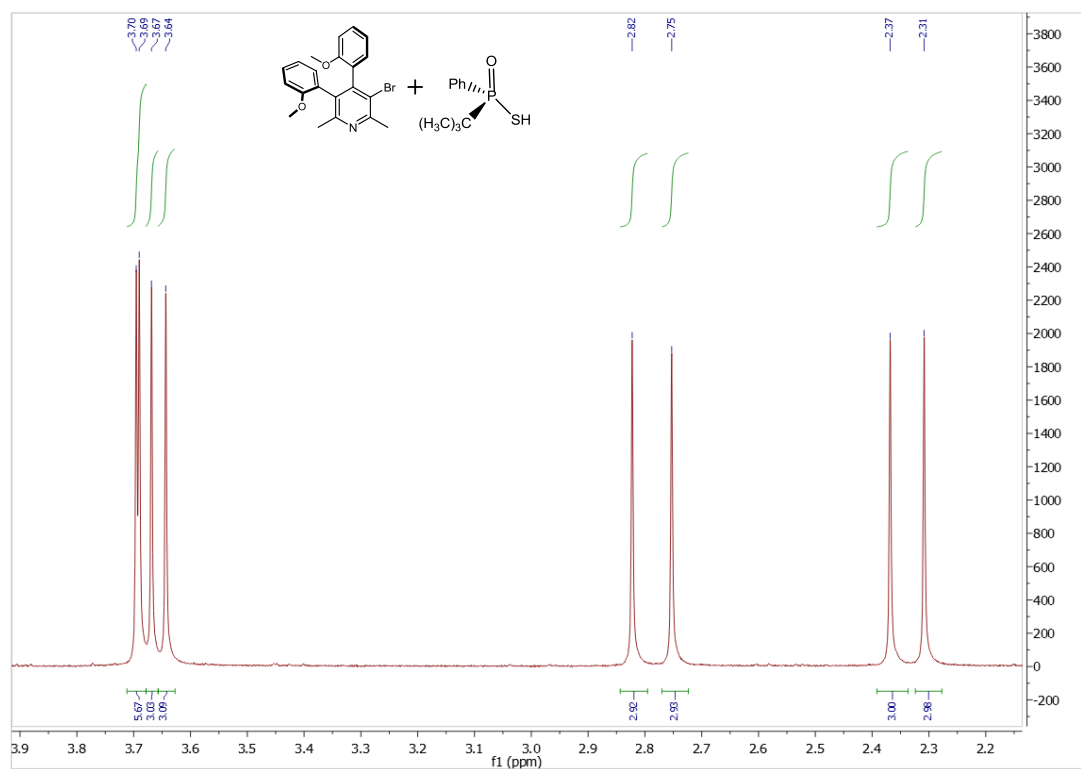
^1H NMR (CH_3 and OCH_3 protons) of racemic **6** in presence of (+)-(*R*)-tert-butyl(phenyl) phosphinothioic acid



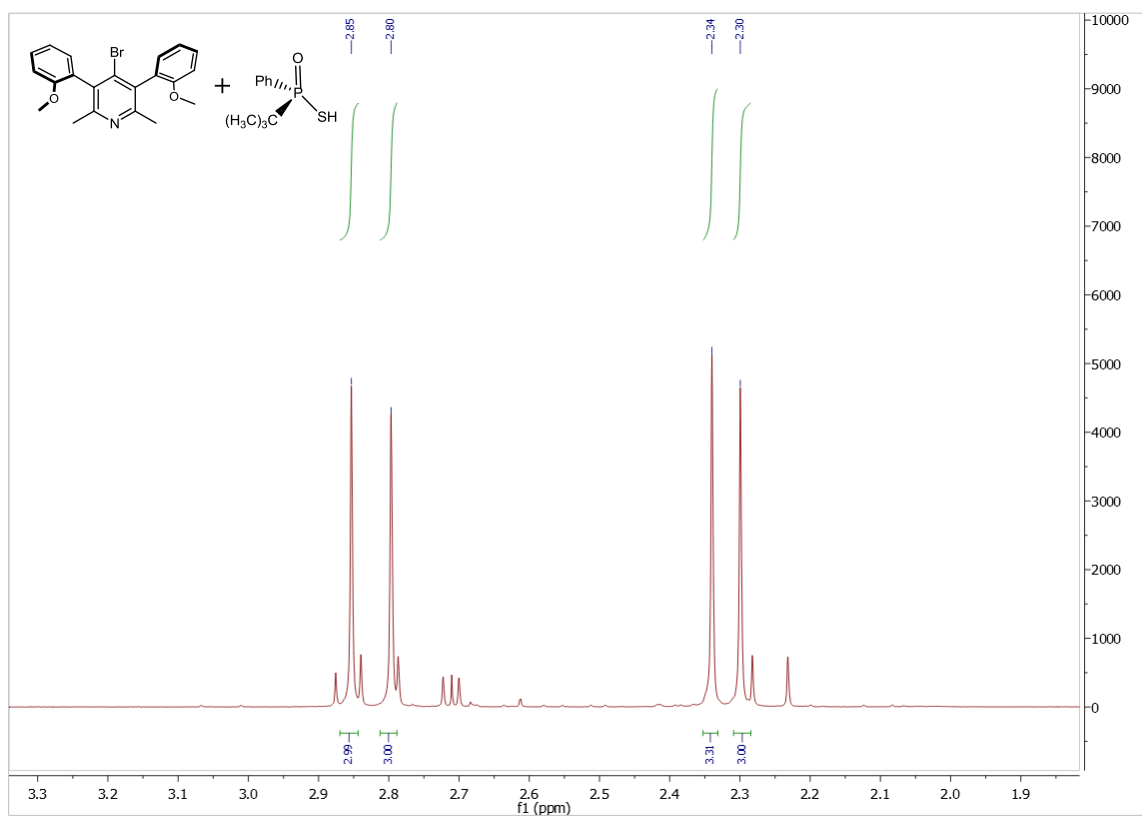
^1H NMR (CH_3 and OCH_3 protons) of racemic **7** in presence of (+)-(*R*)-tert-butyl(phenyl) phosphinothioic acid



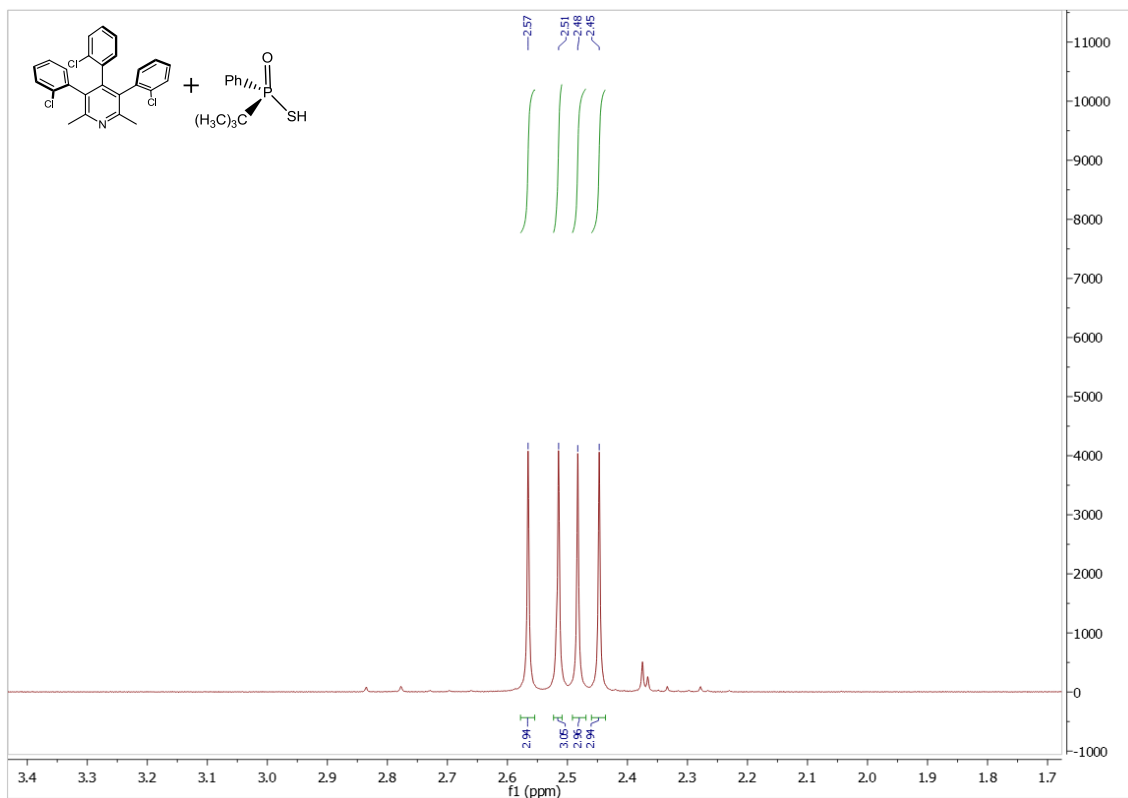
^1H NMR (CH_3 and OCH_3 protons) of racemic **8** in presence of (+)-(*R*)-tert-butyl(phenyl) phosphinothioic acid



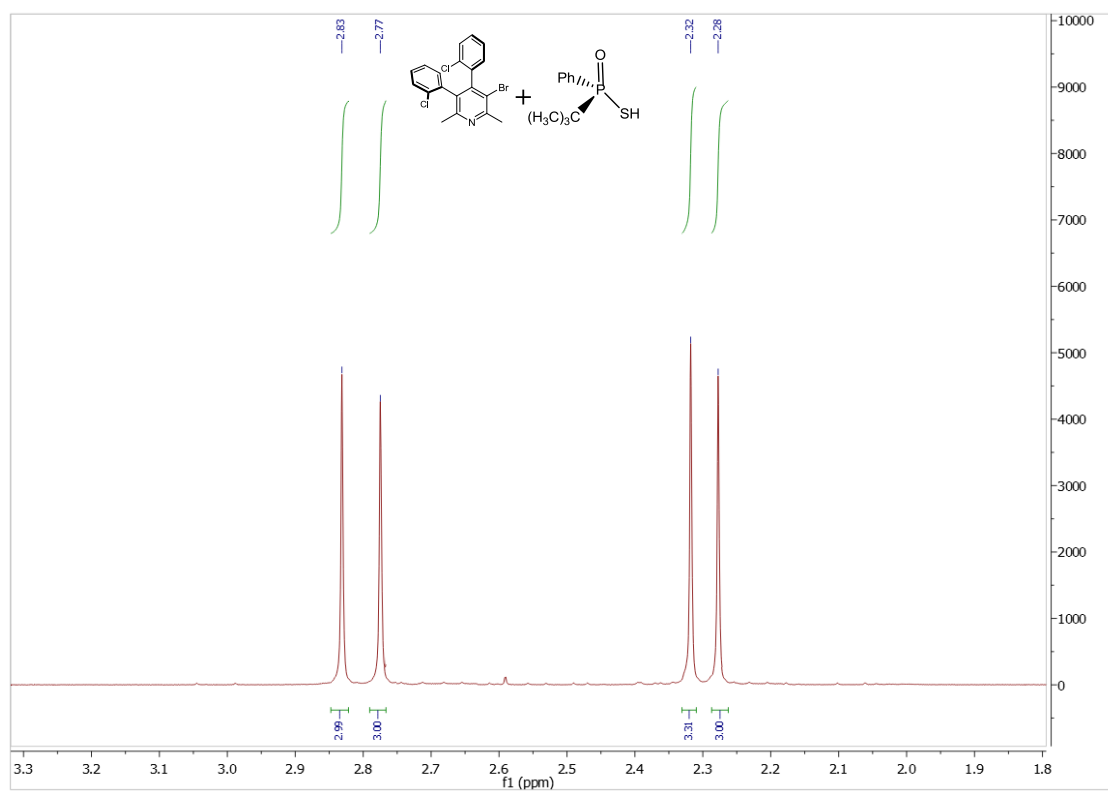
^1H NMR (CH_3 protons) of racemic **9** in presence of (+)-(*R*)-tert-butyl(phenyl) phosphinothioic acid



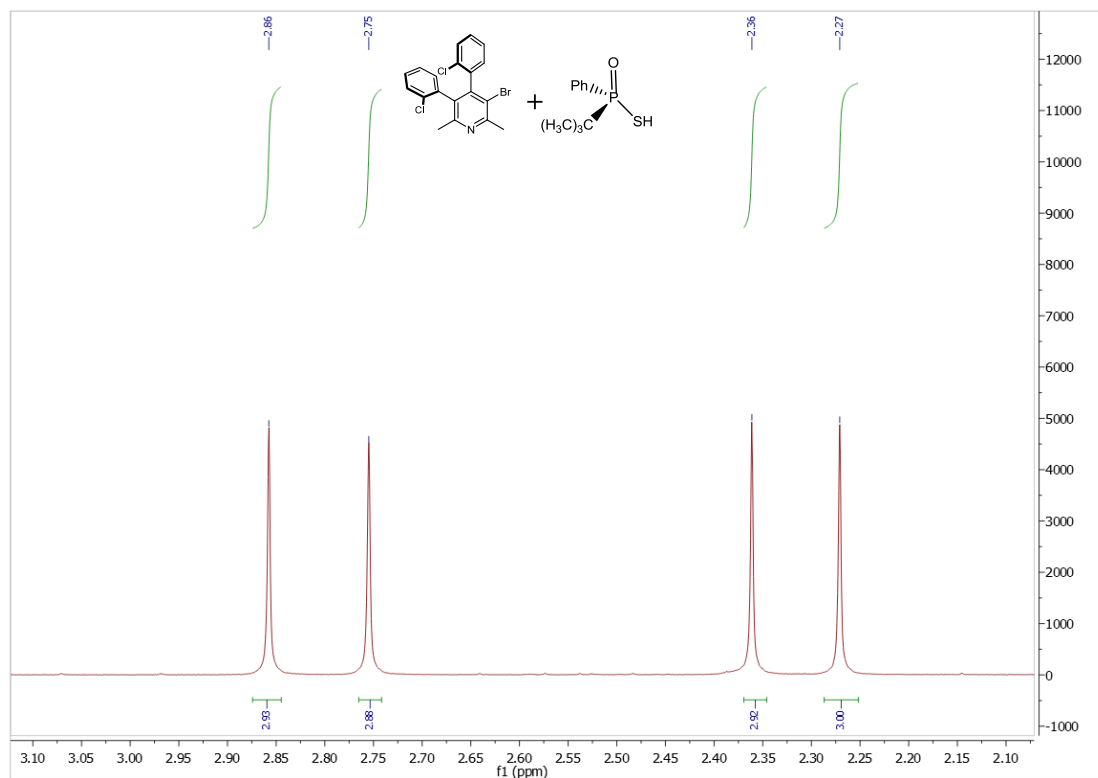
^1H NMR (CH_3 protons) of racemic **13** in presence of (+)-(*R*)-tert-butyl(phenyl)phosphinothioic acid



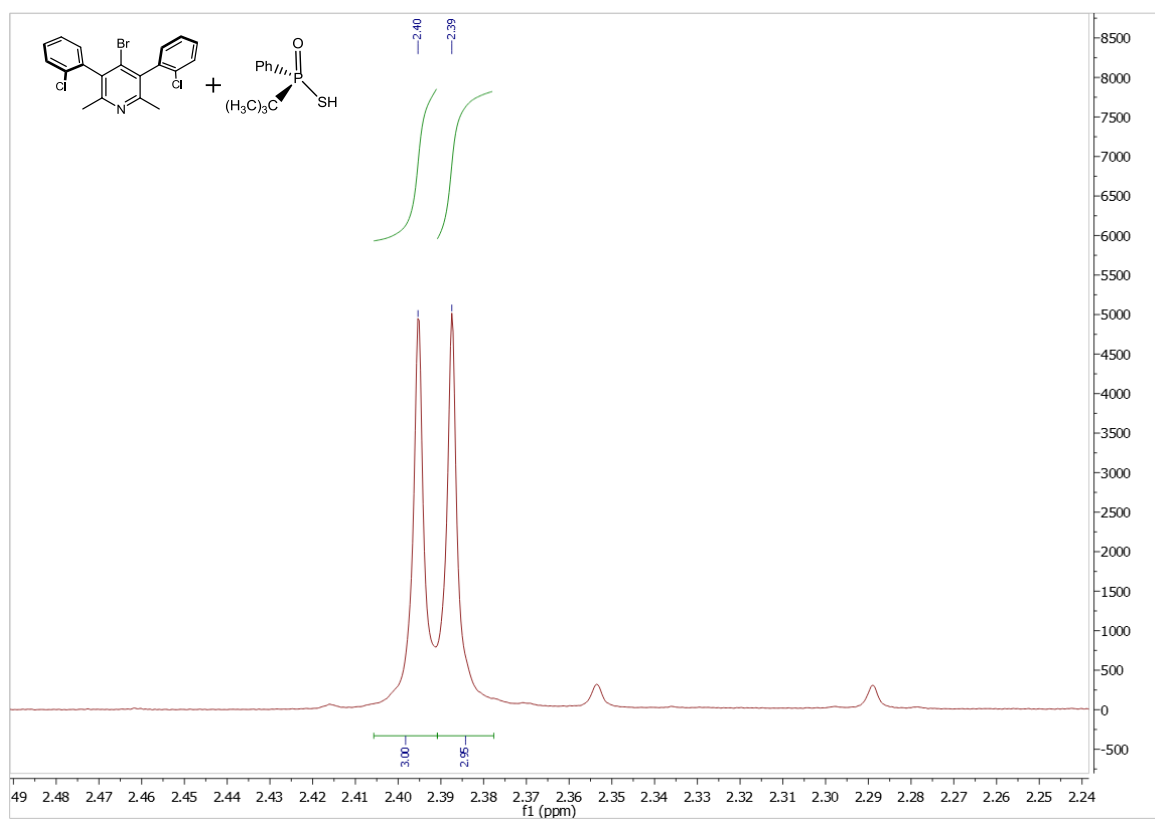
^1H NMR (CH_3 protons) of racemic **15** in presence of (+)-(*R*)-tert-butyl(phenyl)phosphinothioic acid



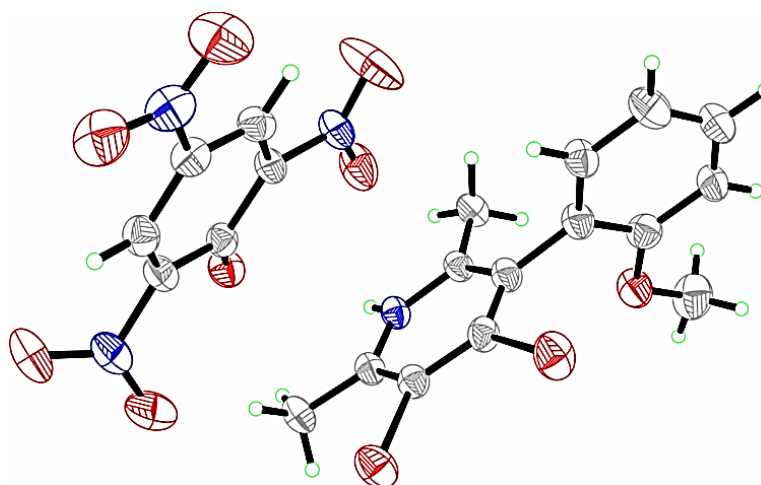
^1H NMR (CH_3 protons) of racemic **16** in presence of (+)-(*R*)-tert-butyl(phenyl)phosphinothioic acid



^1H NMR (CH_3 protons) of racemic **17** in presence of (+)-(*R*)-tert-butyl(phenyl)phosphinothioic acid

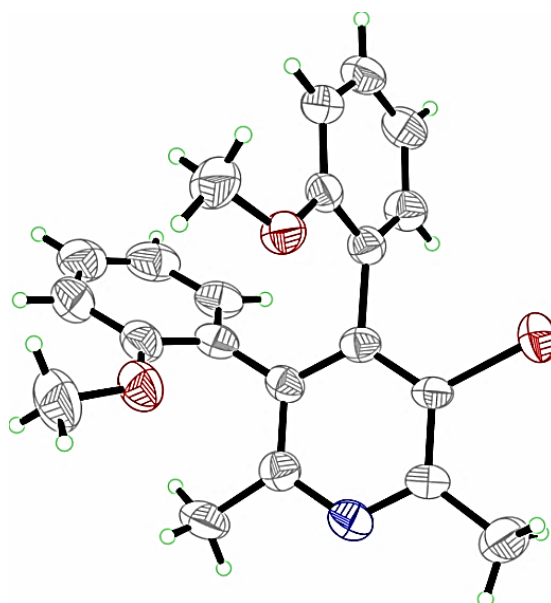


ORTEP diagram of compound **6**



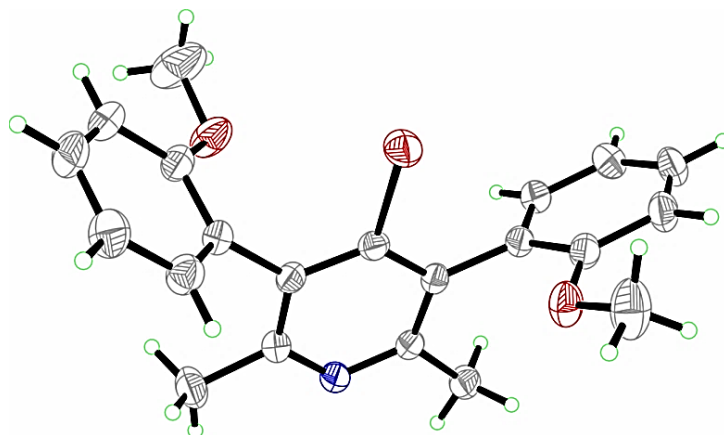
6 * picrate: yellow solid, mp 168.9-169.8°C, crystallization from CH₂Cl₂/hexane
CCDC-1528450 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

ORTEP diagram of compound **7**



White solid, mp 158.5-159.5°C, crystallization from methanol.
CCDC-1528451 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

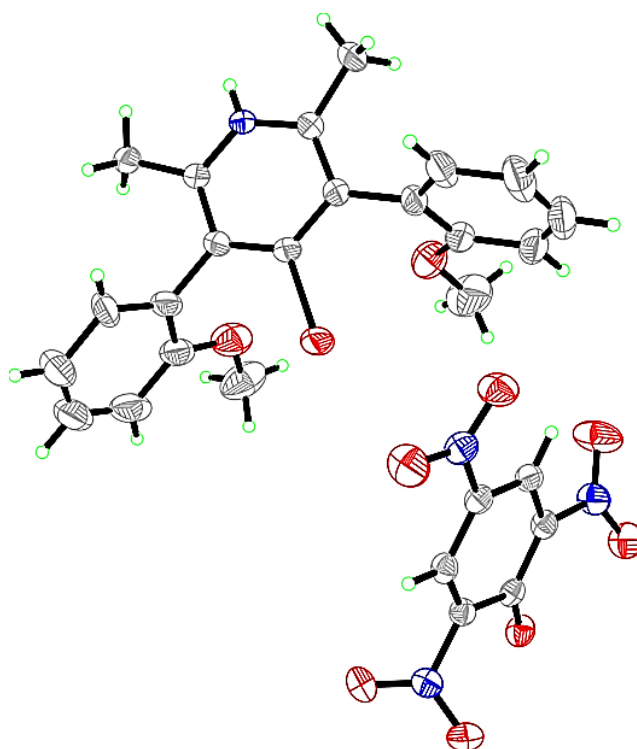
ORTEP diagram of compound **9**



White solid, mp 186.5-187.5 °C, crystallization from methanol.

CCDC-1528454 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

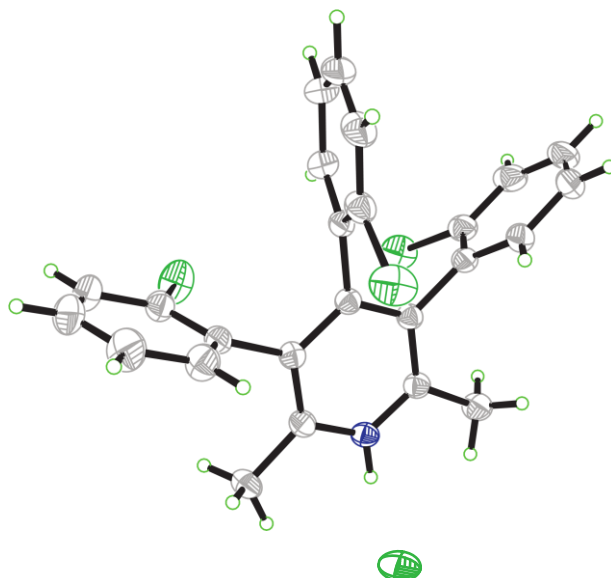
ORTEP diagram of compound **10**



10 * picrate: yellow solid, mp 206.8-207.9°C, crystallization from dichloromethane/hexane.

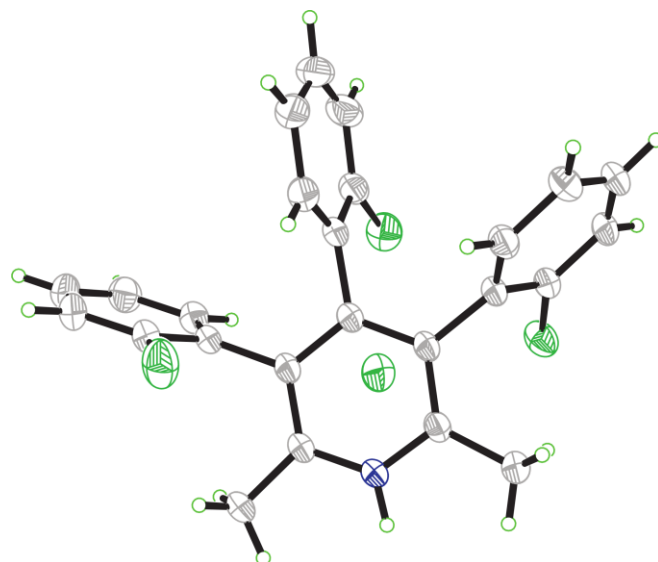
CCDC-1528457 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

ORTEP diagram of compound **12**



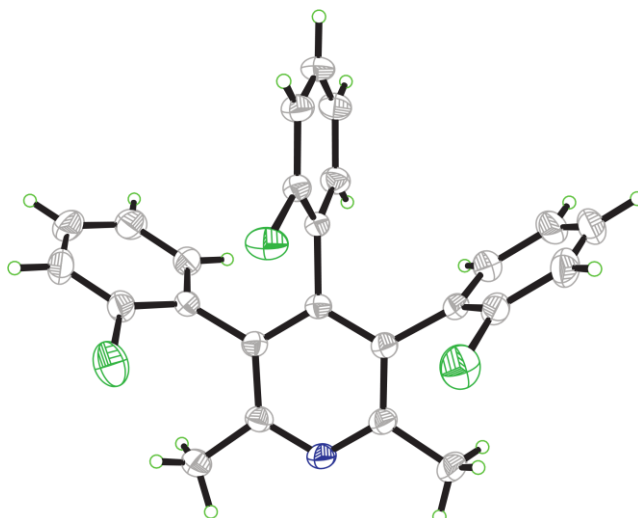
12 * HCl: white solid, mp 233.5-235°C, crystallization from methanol/ethyl acetate
CCDC-1528707 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

ORTEP diagram of compound **13**



13 * HCl: white solid, mp 254-255.5°C, crystallization from dichloromethane/heptane
CCDC-1528679 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

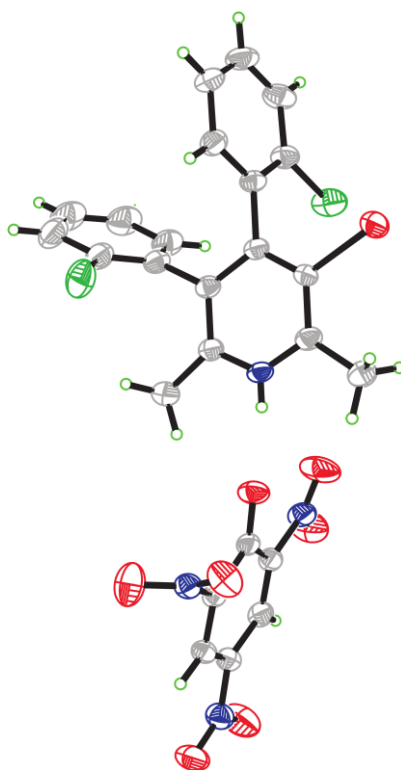
ORTEP diagram of compound **14**



White solid, mp 107-109°C, crystallization from ethyl acetate.

CCDC-1528721 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

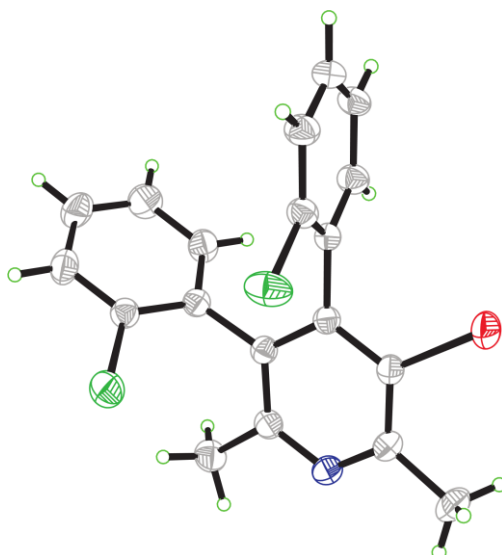
ORTEP diagram of compound **15**



15 * picrate: yellow solid, mp 214-216°C, crystallization from dichloromethane/methanol

CCDC-1528655 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

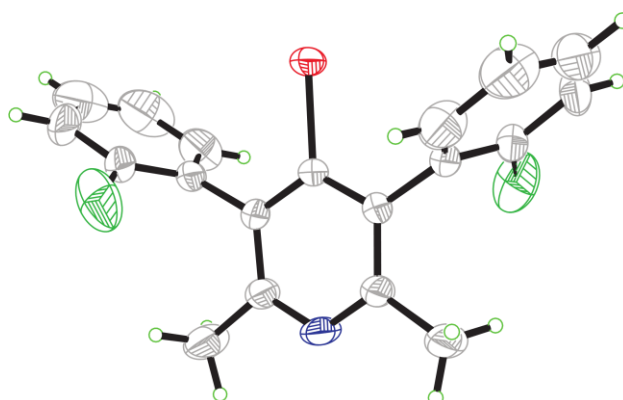
ORTEP diagram of compound **16**



White solid, mp 153-155°C, crystallization from ethyl acetate.

CCDC-1528653 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

ORTEP diagram of compound **17**



White solid, mp 228-230°C, crystallization from ethyl acetate.

CCDC-1528652 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.