

SUPPORTING INFORMATION

SYNTHESIS OF SECONDARY AMIDES FROM THIOCARBAMATES

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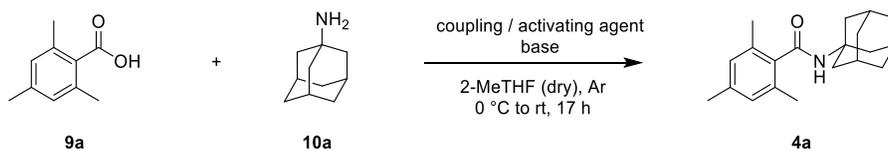
1 General information

Unless stated otherwise, all solvents and commercially available reactants/reagents were used as received. Heptane, which was used for flash chromatography, was distilled prior to use. 2-Methyltetrahydrofuran was dried for 24 h over activated molecular sieves and distilled under nitrogen prior to use. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance 400 MHz spectrometer at 30 °C using the residual solvent as internal standard (^1H : δ 2.50 ppm and ^{13}C [^1H]: δ 39.52 ppm for DMSO- d_6 , ^1H : δ 7.26 ppm and ^{13}C [^1H]: δ 77.16 ppm for CDCl_3 , ^1H : δ 5.98 ppm and ^{13}C [^1H]: δ 73.80 ppm for $\text{C}_2\text{D}_2\text{Cl}_4$). Chemical shifts (δ) are given in ppm and coupling constants (J) are quoted in Hertz (Hz). Multiplicities are described as s (singlet), d (doublet), t (triplet), q (quadruplet), hp (heptuplet), m (multiplet), br (broad) or combinations thereof. ^{13}C NMR spectra were recorded with complete proton decoupling. For high resolution mass-spectrometric analysis, samples were dissolved in methanol or acetonitrile and diluted to a concentration of approximately 10^{-5} mol/L. 2 μL was injected using a CapLC system and electrosprayed through the nanoelectrospray source. The nanoelectrospray source was operated in positive ion mode at an electrospray potential of 1.7 kV. The eluent used was 30% A (H_2O with 0.1% formic acid) and 70% B ($\text{CH}_3\text{CN} / \text{H}_2\text{O}$ 95:5 with 0.1% formic acid) at a flow rate of 6 $\mu\text{L}/\text{min}$. Samples were injected with an interval of 3 min. Before analysis and after each seventh sample a 2 μL volume of a 0.025% H_3PO_4 solution ($\text{MeOH} / \text{H}_2\text{O}$, 50:50) was injected and used as lock mass. The MS was calibrated prior to use with a 0.015% H_3PO_4 solution. The spectra were lock mass corrected using the known mass of the nearest H_3PO_4 cluster. Flash chromatography was performed on an automated chromatography system (Biotage[®] / Combiflash[®]Rf) with on-line UV detection using Silica Flash Cartridges (12 g, Grace[®]) and the indicated solvent system. Melting points were determined by a Büchi Melting Point B-545 device and were uncorrected. The TGA data were obtained on a Thermogravimetric Analyzer TGA2950 (TAInstruments).

2 Classical coupling/activating agents for amide synthesis

Despite the prevalence of reported methods for amide synthesis, the majority is not suitable for preparing challenging amides derived from sterically hindered carboxylic acids and electron-deficient and/or sterically hindered amines. The classical approaches based on coupling/activating agents generally fail for these amides and therefore this remains a challenge in the field of amide synthesis. This is illustrated for the synthesis of *N*-(1-adamantyl)-2,4,6-trimethylbenzamide (**4a**, Table S1) and *N*-mesityl-2,4,6-trimethylbenzamide (**4k**, Table S2) from 2,4,6-trimethylbenzoic acid (**9a**) and 1-adamantylamine (**10a**) or 2,4,6-trimethylaniline (**10k**), respectively. A variety of classical coupling agents (Scheme S1) were tested under different conditions, but generally no reaction product was formed, only with HATU **3a** could be obtained in a low yield (Table S1, entry 4). When PyBOP was employed for the synthesis of **4k**, the desired amide was isolated only in a moderate yield (Table S2, entry 5).

Table S1. Screening of Classical Coupling / Activating agents for the Synthesis of *N*-(1-Adamantyl)-2,4,6-trimethylbenzamide (4a**) from 2,4,6-Trimethylbenzoic acid (**9a**) and 1-Adamantylamine (**10a**)^a**



| entry | 9a (equiv) | 10a (equiv) | coupling / activating agent (equiv) | base (equiv) | yield 4a (%) ^b | reference |
|----------------|-------------------|--------------------|-------------------------------------|---|----------------------------------|-----------|
| 1 ^c | 1.0 | 1.0 | SOCl ₂ (1.20) | NaHCO ₃ (1.00) | 0 | 1 |
| 2 | 1.0 | 1.0 | DCC (1.08) | DMAP (0.25) | 0 | 2 |
| 3 | 1.0 | 1.2 | DCC (1.00) | HOBt (1.10) | 0 | 2 |
| 4 | 1.0 | 1.0 | HATU (1.50) | Et ₃ N (3.00) | 18 | 3 |
| 5 | 1.0 | 1.1 | COMU (1.20) | EtN(ⁱ Pr) ₂ (2.00) | 0 | 4 |
| 6 | 1.0 | 1.2 | T ₃ P (1.50) | EtN(ⁱ Pr) ₂ (2.00) | 0 | 4 |
| 7 ^d | 1.0 | 1.0 | CDI (1.05) | DBU (0.50) | 0 | 5 |

^a Reaction conditions: 2,4,6-trimethylbenzoic acid (**9a**, 82 mg, 0.5 mmol), 1-adamantylamine (**10a**), coupling agent, base, 2-methyltetrahydrofuran (2.0 mL, dry), argon, 0 °C to rt, 17 h. ^b ¹H NMR yield using 1,3,5-trimethoxybenzene as internal standard. ^c Acyl chloride formation prior to the addition of 1-adamantylamine (**10a**, 0.5 mmol, 1.0 equiv), NaHCO₃ (42 mg, 0.5 mmol, 1.0 equiv) in diethyl ether under air. ^d Reaction at 80 °C.

DMAP = 4-dimethylaminopyridine. HOBt = hydroxybenzotriazole. DBU = 1,8-diazabicyclo[4.5.0]undec-7-ene.

Scheme S1. Example of Classical Amide Coupling Agents for Amide Synthesis and Their Health, Flammability and Reactivity (HFR) rating Scale from 0 (low) to 4 (high).

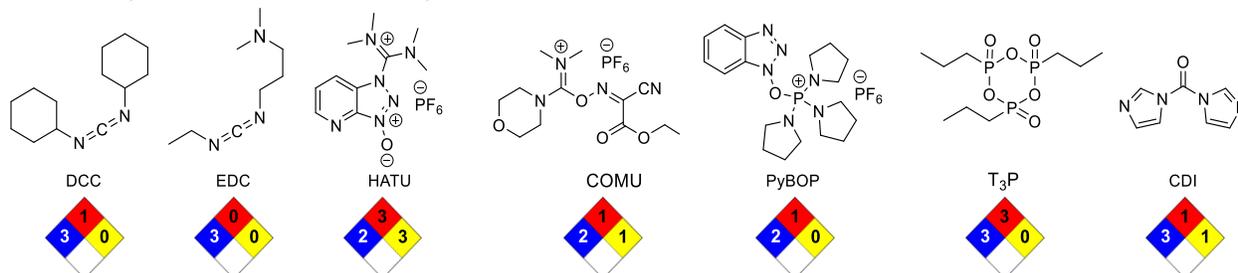
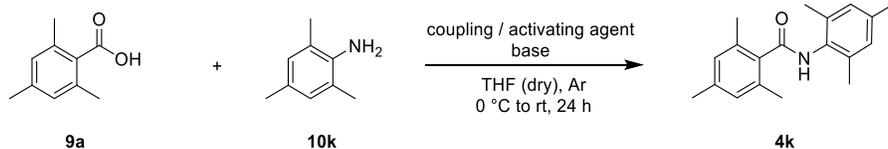


Table S2. Screening of Classical Coupling Agents for the Synthesis of *N*-Mesityl-2,4,6-trimethylbenzamide (4k**) from 2,4,6-Trimethylbenzoic acid (**9a**) and 2,4,6-Trimethylaniline (**10k**)^a**

| entry | 9a (equiv) | 10k (equiv) | coupling / activating agent (equiv) | base (equiv) | yield 4k (%) ^b | reference |
|----------------|-------------------|--------------------|-------------------------------------|---|----------------------------------|-----------|
| 1 ^c | 1.0 | 1.0 | DCC (1.08) | DMAP (0.25) | 0 | 2 |
| 2 | 1.0 | 1.2 | DCC (1.00) | HOBt (1.10) | 0 | 2 |
| 3 | 1.0 | 1.0 | EDC (1.20) | HOBt (0.20) / EtN(ⁱ Pr) ₂ (2.00) | 0 | 4 |
| 4 | 1.0 | 1.0 | HATU (1.50) | EtN(ⁱ Pr) ₂ (2.00) | Trace | 4 |
| 5 | 1.0 | 1.0 | PyBOP (1.50) | EtN(ⁱ Pr) ₂ (2.00) | 61 | 4 |
| 6 | 1.0 | 1.2 | T ₃ P (1.50) | EtN(ⁱ Pr) ₂ (2.00) | 0 | 4 |
| 7 | 1.0 | 1.0 | CDI (1.05) | / | 0 | 4 |
| 8 | 1.0 | 1.0 | CDI (1.05) | DBU (0.50) | 0 | 5 |
| 9 | 1.0 | 1.0 | DCC (1.08) | DMAP (0.25) | 0 | 2 |

^a Reaction conditions: 2,4,6-trimethylbenzoic acid (**9a**, 82 mg, 0.5 mmol), 2,4,6-trimethylaniline (**10k**), coupling agent, base, tetrahydrofuran (2.0 mL, dry), argon, 0 °C to rt, 24 h. ^b Isolated yield. ^c Dichloromethane (2.0 mL, dry) as solvent.

DMAP = 4-dimethylaminopyridine. HOBt = hydroxybenzotriazole. DBU = 1,8-diazabicyclo[4.5.0]undec-7-ene.

3 Thermogravimetric analysis (TGA)

The group of Bode reported a strategy for the synthesis of secondary amides based on the addition of Grignard reactants to isocyanates⁶ or carboxyanhydrides (NCAs).⁷ Therefore the stability of thiocarbamates **1a**, **1b** and **8** was compared with the corresponding isocyanates **11a**, **11b** and **11c**, using thermogravimetric analysis (TGA) (Figure S1-Figure S4). The following method was applied: starting temperature: 40 °C, heating: 10 °C / min to 350 °C under the indicated atmosphere. However, no difference in weight loss is observed under nitrogen or air atmosphere (Figure S1 and Figure S2). For all examined thiocarbamates, the decomposition temperature is higher than their melting point (Table S3; Figure S1; Figure S3-Figure S4) In addition, these TGA analysis illustrate that in each case the thiocarbamate is more stable than its corresponding isocyanate. For phenyl isocyanate (**11c**) a weight loss is observed between 70 and 100 °C (Figure S4) although its boiling point is 162-163 °C (Table S3, entry 6 and Figure S5). Similarly, no TGA could be recorded for *tert*-butyl isocyanate (**11b**) as all product was evaporated at the start of the analysis (40 °C). These results demonstrate that the thiocarbamates are more stable compounds and are easier to handle than the isocyanates.

Table S3. Experimental Melting and/or Boiling Points of Thiocarbamates and Isocyanates.

| entry | thiocarbamate or isocyanate | melting point (°C) | boiling point (°C) |
|-------|--|--------------------|--------------------|
| 1 | <i>S</i> -phenyl (1-adamantyl)thiocarbamate (1a) | 137-138 | / |
| 2 | 1-Adamantylisocyanate (11a) | 144-145 | / |
| 3 | <i>S</i> -phenyl <i>tert</i> -butylthiocarbamate (1b) | 114-115 | / |
| 4 | <i>tert</i> -butyl isocyanate (11b) | / | 85-86 |
| 5 | <i>N,S</i> -diphenylthiocarbamate (8) | 120-121 | / |
| 6 | Phenylisocyanate (11c) | -30 | 162-163 |

Figure S1. Thermogravimetric Analysis of *S*-phenyl (1-adamantyl)thiocarbamate (1a**, left) and 1-Adamantylisocyanate (**11a**, right) under Nitrogen Atmosphere**

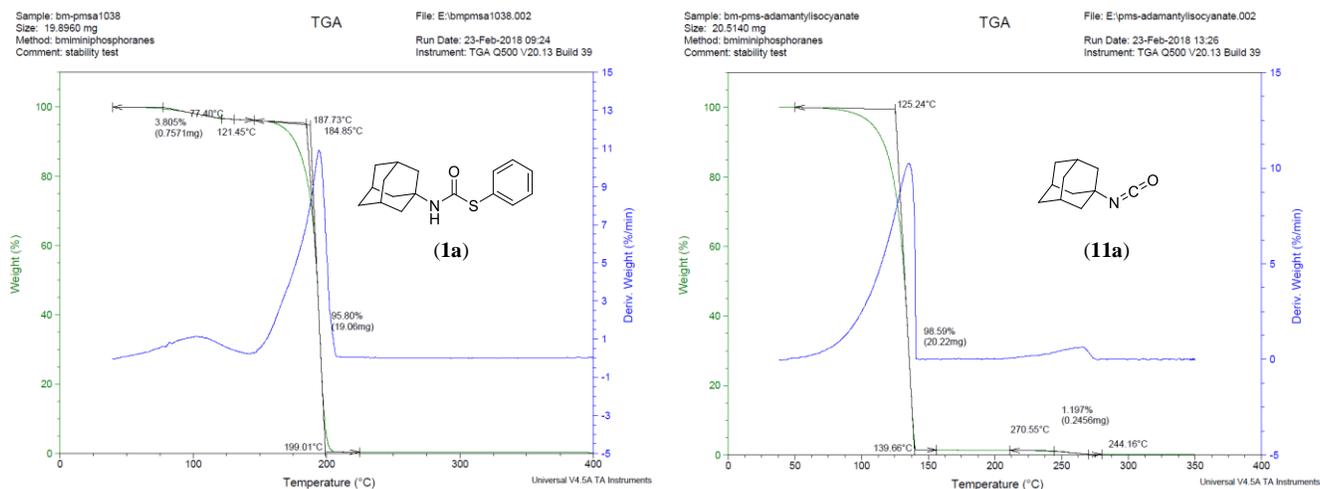


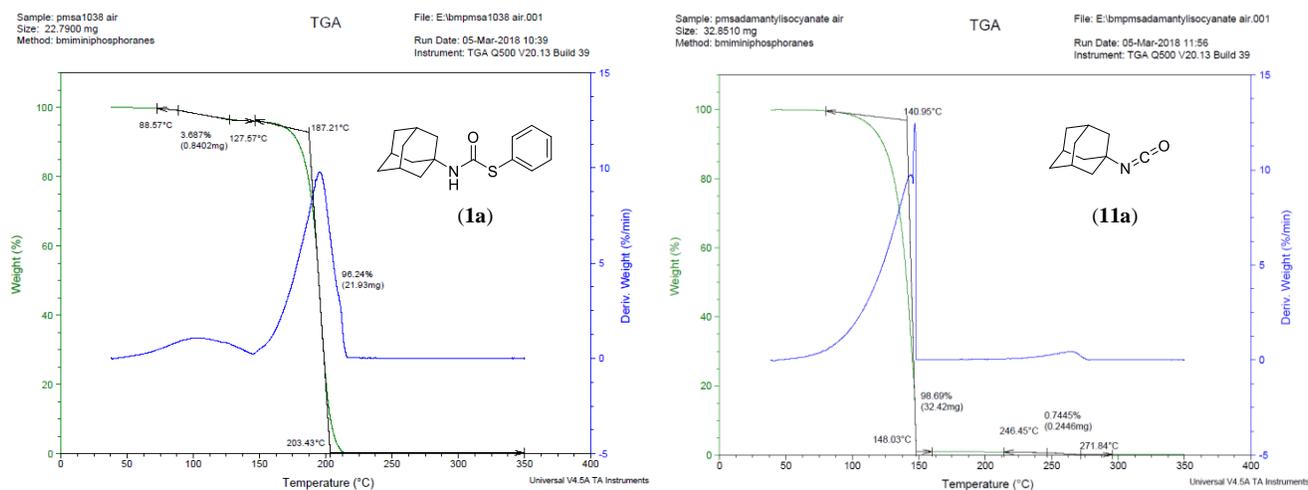
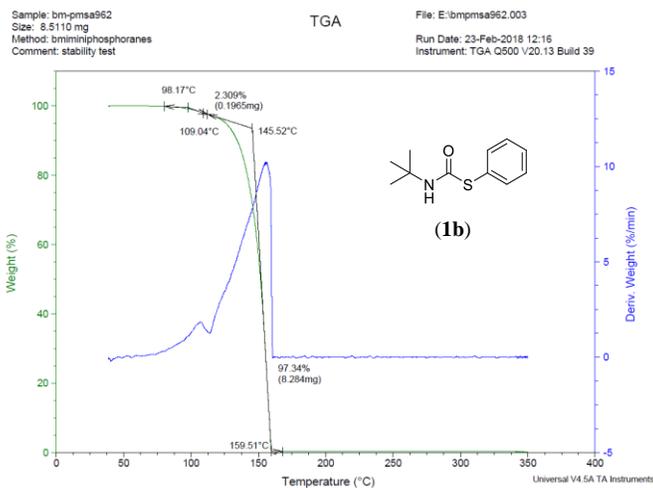
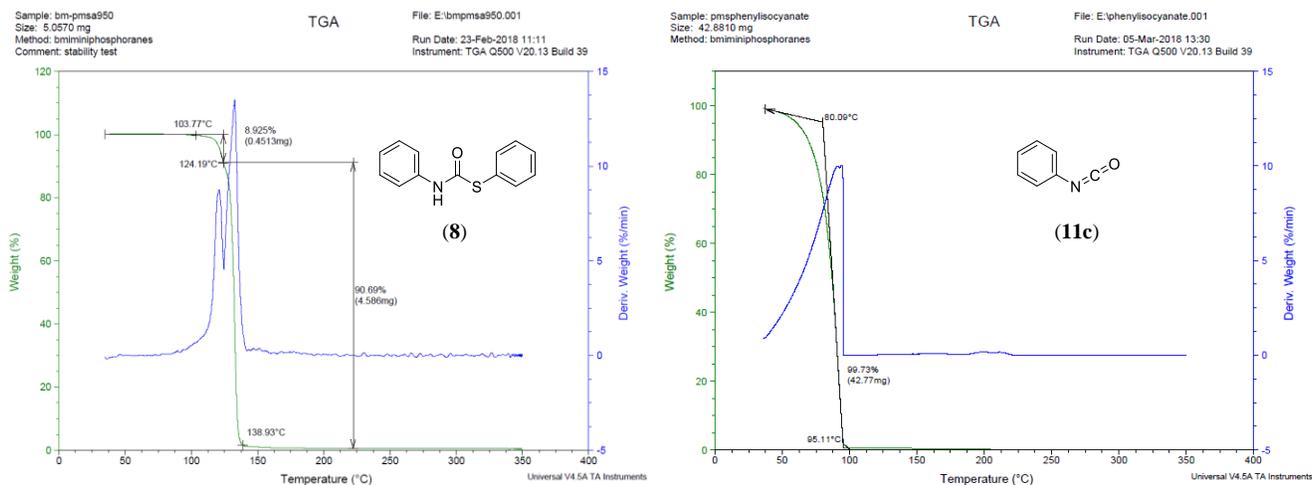
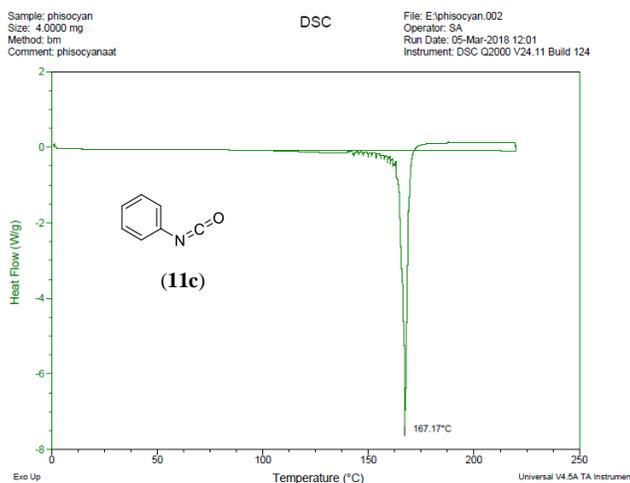
Figure S2. Thermogravimetric Analysis of *S*-Phenyl (1-adamantyl)thiocarbamate (1a, left) and 1-Adamantylisocyanate (11a, right) under Air Atmosphere.

Figure S3: Thermogravimetric Analysis of *S*-Phenyl *tert*-butylthiocarbamate (1b) under Nitrogen Atmosphere.

Figure S4: Thermogravimetric Analysis of *N,S*-Diphenylthiocarbamate (8, left) and Phenyl Isocyanate (11c, right) under Nitrogen Atmosphere.


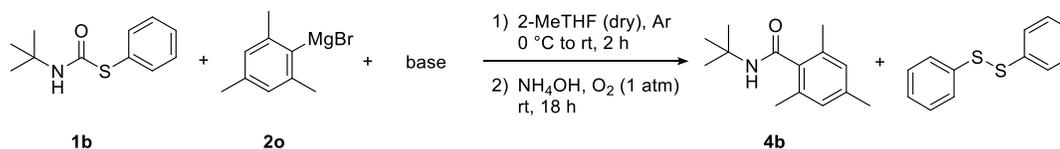
Figure S5: Differential Scanning Calorimetry of Phenyl Isocyanate (11c)



4 Search for an alternative sacrificial base

Our methodology towards secondary amides requires two equivalents of the Grignard reactant of which one acts as a sacrificial base. When it is derived from an expensive alkyl or aryl bromide it would be better to be able to use a cheaper base instead. The synthesis of **4b** was selected as a model reaction to check the feasibility of premixing several bases with one equivalent of mesitylmagnesium bromide (**2o**) prior to the addition of thiocarbamate **1b** (Table S4). 1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU) and potassium bis(trimethylsilyl)amide (KHMDS) are not suitable for this purpose as **4b** was not obtained (entries 2-3). *N*-(*tert*-Butyl)-2,4,6-trimethylbenzamide (**4b**) was obtained in low yield when organolithium reactants were used as sacrificial base as exemplified by ⁿBuLi and ^tBuLi (entries 4-5). In addition, also substantial amounts of undesired *N*-*tert*-butylpentanamide and *N*-*tert*-butylpivalamide (**3m**) were respectively obtained due to the competitive action of the base as a nucleophile. Pleasingly, with one equivalent of readily available ^tBuMgCl as a sacrificial base, *N*-(*tert*-butyl)-2,4,6-trimethylbenzamide (**4b**) was isolated in high yield without the formation of *N*-*tert*-butylpivalamide (**3m**) as a side product (entry 6).

Table S4: Reaction of mesitylmagnesium bromide (**2o**) with *S*-phenyl *tert*-butylthiocarbamate (**1b**) using a sacrificial base^a



| entry | base | yield 4b (%) ^b | yield PhSSPh (%) ^b |
|-------|---------------------|----------------------------------|-------------------------------|
| 1 | 2o | 92 | 91 |
| 2 | DBU | 0 | 71 |
| 3 | KHMDS | 0 | 74 |
| 4 | ⁿ BuLi | 34 ^c | 46 |
| 5 | ^t BuLi | 24 ^d | 44 |
| 6 | ^t BuMgCl | 85 | 81 |

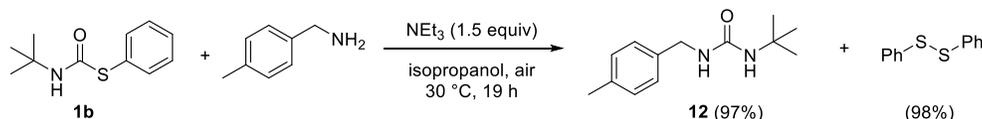
^a Reaction conditions: *S*-phenyl *tert*-butylthiocarbamate (**1b**, 52 mg, 0.25 mmol), 2,4,6-trimethylphenylmagnesium bromide (**2o**, 275 μ L, 0.28 mmol, 1.1 equiv), base (0.25 mmol, 1.0 equiv), 2-methyltetrahydrofuran (2 mL, dry), argon, 0 °C to rt, 2 h. Then NH₄OH (3 mL), O₂ (balloon, 1 atm), rt, 18 h. Isolated yield. ^b ¹H NMR yield using 1,3,5-trimethoxybenzene as internal standard. ^c *N*-*tert*-Butylpentanamide was obtained in 52% yield. ^d *N*-*tert*-Butylpivalamide (**3m**) was obtained in 22% yield.

DBU = 1,8-diazabicyclo[5.4.0]undec-7-ene. KHMDS = potassium bis(trimethylsilyl)amide.

5 Reaction of *S*-phenyl thiocarbamates with amines

Besides the straightforward transformation of thiocarbamates into amides (this work) and carbamates (our previous work),⁸ urea are also easily obtained (Scheme S2). No waste resulting from the thiophenol leaving group is generated as it can easily be recovered as diphenyl disulfide, which can be transformed afterwards into *S*-phenyl benzenethiosulfonate by selective oxidation.⁹ The transformation of *S*-aryl thiocarbamates into ureas has been reported for specific examples in acetonitrile at 30 °C.¹⁰ A large excess (e.g. more than 10 equivalents) of amine was required to achieve the aminolysis and the expelled *S*Ar group could not be easily recovered. However, in this work urea **12** is obtained in high yield in isopropanol via a reaction of **1b** with 4-methylbenzylamine, even at 30 °C, with almost quantitative recovery of the leaving group. The oxidant for disulfide formation is provided by simply performing the reaction under air.

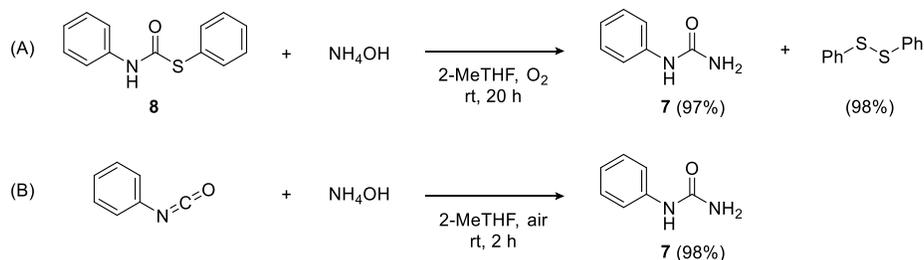
Scheme S2. Urea Synthesis from *S*-phenyl *tert*-butylthiocarbamate (**1b**)



Reaction conditions: *S*-phenyl *tert*-butylthiocarbamate (**1b**, 105 mg, 0.5 mmol), 4-methylbenzylamine (64 μ L, 0.5 mmol), NEt₃ (105 μ L, 0.75 mmol, 1.5 equiv), isopropanol (1.0 mL), 30 °C, 19 h, air.

When phenylmagnesium bromide (**2f**), *tert*-butylmagnesium bromide (**2m**), *sec*-butylmagnesium bromide (**2n**) or *n*-butylmagnesium bromide (**2p**) were coupled with *N,S*-diphenylthiocarbamate (**8**) surprisingly only low yields of amides **6a**, **6c-6e** were obtained (Scheme 4, manuscript), although diphenyl disulfide was isolated in good yield. Instead, *N*-phenyl urea (**7**) was obtained. Both diphenyl disulfide and **7** are formed by decomposition of the thiocarbamate substrate with ammonium hydroxide during the workup (Scheme S3). This probably occurs via a phenyl isocyanate intermediate followed by ammonia addition to generate *N*-phenylurea (**7**).

Scheme S3. Reaction of *N,S*-diphenylthiocarbamate (**8**) or Phenyl Isocyanate with Ammonia Solution



Reaction conditions: (A) *S*-phenyl *tert*-butylthiocarbamate (**8**, 115 mg, 0.5 mmol), 2-MeTHF (2 mL), NH₄OH (2 mL), rt, 20 h, O₂ (1 atm). (B) phenyl isocyanate (54 μ L, 0.5 mmol, 1.0 equiv), 2-MeTHF (2 mL), NH₄OH (2 mL), rt, 2 h, air.

6 Experimental

6.1 Synthesis of thiocarbamates

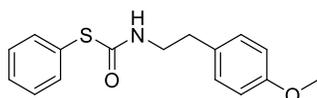
The synthesis and characterization of *S*-phenyl (1-adamantyl)thiocarbamate (**1a**), *S*-phenyl *tert*-butylthiocarbamate (**1b**), *S*-phenyl (2,4,4-trimethylpentan-2-yl)thiocarbamate (**1c**), methyl 2-methyl-2-[(phenylthio)carbonyl]amino}propanoate (**1d**), *S*-phenyl cyclohexylthiocarbamate (**1e**), *S*-phenyl (1-phenylethyl)thiocarbamate (**1f**), *S*-phenyl benzylthiocarbamate (**1g**), *S*-phenyl pentylthiocarbamate (**1h**), *S*-phenyl 2,6-dimethylphenylthiocarbamate (**1j**), *S*-phenyl (4-methoxyphenyl)thiocarbamate (**1m**), has been previously described by us.⁸ *N,S*-diphenylthiocarbamate (**8**) has been prepared according to Movasagh *et al.*¹¹

Compounds *S*-phenyl 4-methoxyphenylethylthiocarbamate (**1i**), *S*-phenyl 2,4,6-trimethylphenylthiocarbamate (**1k**), *S*-phenyl (2,6-diisopropylphenyl)thiocarbamate (**1l**) have been prepared by our previously developed method.⁸

General procedure:⁸

A 10 mL round-bottom flask was charged with sodium iodide (5 mol%), *S*-aryl/alkyl benzenethiosulfonate (1.0 equiv), isocyanide (1.2 equiv) and isopropanol (0.5 mL, 6.5 equiv). The reaction mixture was stirred under air in a preheated oil bath at 30 °C for 4 h. Subsequently, the solvent was removed under reduced pressure and the product was purified by an automated flash chromatography system using silica cartridges and the eluent indicated below.

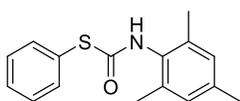
S-phenyl 4-methoxyphenylethylthiocarbamate (**1i**) [PMSA-CVE013/015]



The general procedure was applied using *S*-phenyl benzenethiosulfonate (626 mg, 2.5 mmol, 1.0 equiv) and 1-(2-isocyanoethyl)-4-methoxybenzene (484 mg, 3.0 mmol, 1.2 equiv) and isopropanol (4.0 mL). The reaction mixture was stirred for 15 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 25% ethyl acetate, 25 mL/min). *S*-Phenyl 4-methoxyphenylethylthiocarbamate was obtained in 94% (672 mg) yield. Isopropyl benzenesulfonate was isolated in 88% (404 mg) yield. New compound according to Scifinder.

Light yellow solid, m.p.: 77-78 °C, $R_f = 0.12$ in heptane / EtOAc (9:1), ¹H NMR (400 MHz, CDCl₃): δ 7.51 - 7.49 (m, 2H), 7.41 - 7.35 (m, 3H), 7.02 - 7.00 (d, $J = 7.9$ Hz, 2H), 6.82 - 6.80 (d, $J = 8.4$ Hz, 2H), 5.29 (br s, 1H), 3.80 (s, 3H), 3.46 (dd, $J = 12.8, 6.5$ Hz, 2H), 2.72 (t, $J = 6.5$ Hz, 2H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 166.3 (C), 158.5 (C), 135.6 (CH), 130.4 (C), 129.8 (CH), 129.8 (CH), 129.6 (CH), 128.7 (C), 114.3 (CH), 55.4 (CH₃), 42.8 (CH₂), 34.7 (CH₂) ppm. HRMS (ESI) for C₁₆H₁₈NO₂S [M+H]⁺ calcd. 288.1053, found 288.1065.

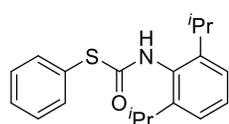
S-phenyl 2,4,6-trimethylphenylthiocarbamate (**1k**) [PMSA1026]



The general procedure was applied using *S*-phenyl benzenethiosulfonate (250 mg, 1.0 mmol, 1.0 equiv) and 2,4,6-dimethylphenylisocyanide (218 mg, 1.2 mmol, 1.2 equiv). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 20% ethyl acetate, 25 mL/min). *S*-phenyl 2,4,6-trimethylphenylthiocarbamate was obtained in 85% (287 mg) yield. New compound according to Scifinder. Due to the existence of rotamers, the NMR spectra were recorded at 100 °C in C₂D₂Cl₄.

White solid, m.p.: 154-155 °C, ¹H NMR (400 MHz, C₂D₂Cl₄): δ 7.67 - 7.64 (m, 2H), 7.47 - 7.44 (m, 3H), 6.95 (s, 2H), 6.53 (br s, 1H), 2.34 (s, 3H), 2.29 (s, 6H) ppm. ¹³C NMR (101 MHz, C₂D₂Cl₄): δ 165.5 (br, C), 137.7 (C), 135.9 (C), 135.0 (CH), 130.9 (C), 129.2 (CH), 129.1 (C), 129.1 (CH), 128.9 (CH), 20.6 (CH₃), 17.84 (CH₃) ppm. HRMS (ESI) for C₁₆H₁₈NOS [M+H]⁺ calcd. 272.1104, found 272.1118.

S-phenyl (2,6-diisopropylphenyl)thiocarbamate (**1l**) [PMSA998-CVE]



The general procedure was applied using *S*-phenyl benzenethiosulfonate (250 mg, 1.0 mmol, 1.0 equiv) and 2,6-diisopropylphenylisocyanide (225 mg, 1.2 mmol, 1.2 equiv). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 5% ethyl acetate, 25 mL/min). *S*-Phenyl (2,6-diisopropylphenyl)thiocarbamate was obtained in 85% (266 mg) yield. New compound according to Scifinder. Due to the existence of rotamers, the NMR spectra were recorded at 100 °C in C₂D₂Cl₄.

White solid, m.p.: 167-169 °C, $R_f = 0.22$ in heptane / EtOAc (9:1), $^1\text{H NMR}$ (400 MHz, $\text{C}_2\text{D}_2\text{Cl}_4$): δ 7.63 (s, 2H), 7.45 - 7.44 (m, 3H), 7.34 (t, $J = 7.7$ Hz, 1H), 7.20 (d, $J = 7.7$ Hz, 2H), 6.54 (br s, 1H), 3.23 - 3.20 (m, 2H), 1.28 (d, $J = 6.6$ Hz, 12H) ppm. $^{13}\text{C NMR}$ (101 MHz, $\text{C}_2\text{D}_2\text{Cl}_4$): δ 146.9 (C), 135.0 (CH), 130.7 (CH), 129.2 (CH), 129.1 (CH), 128.9 (CH), 123.5 (CH), 28.7 (CH), 23.3 (CH_3) ppm. HRMS (ESI) for $\text{C}_{19}\text{H}_{24}\text{NOS}$ $[\text{M}+\text{H}]^+$ calcd. 314.1573, found 314.1574.

6.2 Synthesis of secondary amides

General Procedure A:

A Schlenk flask was flame-dried under vacuum and cooled to room temperature under an argon flow. Subsequently, thiocarbamate (**1**, 0.5 mmol) and 2-methyltetrahydrofuran (2.0 mL, dry) were added and the solution was cooled to 0 °C. Subsequently, the Grignard reactant (**2**, 1.1 mmol, 2.2 equiv) was added slowly over 5 min under an argon atmosphere and slowly warmed to room temperature. After stirring for 2 h, the mixture was quenched with ammonia solution (NH_4OH , 25% in H_2O , 3 mL). Oxygen was bubbled through the solution for 30 min and the mixture was stirred further for 18 h under oxygen atmosphere (balloon). The 2-methyltetrahydrofuran was evaporated, water (5 mL) was added and the mixture was extracted with ethyl acetate (3 x 10 mL). The combined organic phases were dried over anhydrous MgSO_4 , filtered and concentrated *in vacuo*. The product was purified by an automated flash chromatography system using silica cartridges and the indicated solvent.

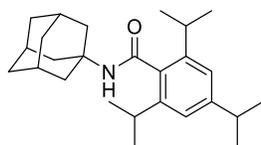
General procedure B:

A Schlenk flask was flame-dried under vacuum and cooled to room temperature under an argon flow. Under argon magnesium turnings (15 mg, 0.55 mmol, 2.2 equiv), a crystal of iodine and 2-methyltetrahydrofuran (2.0 mL, dry) were added. Subsequently, the halogenated substrate (0.55 mmol, 2.2 equiv) was added in one portion. When the Grignard formation started, the reaction mixture was heated at 60 °C for 1 h. Afterwards, the mixture was cooled to 0 °C and a solution of the thiocarbamate (**1**, 0.25 mmol) in 2-methyltetrahydrofuran (2.0 mL, dry) was added dropwise over 5 min. The reaction mixture was slowly warmed to room temperature and stirred for 2 h. The mixture was quenched with ammonia solution (NH_4OH , 25% in H_2O , 3 mL). Oxygen was bubbled through the solution for 30 min and the mixture was stirred further for 18 h under oxygen atmosphere (balloon). The 2-methyltetrahydrofuran was evaporated, water was added (5 mL) and the mixture was extracted with ethyl acetate (3 x 10 mL). The combined organic phases were dried over anhydrous MgSO_4 , filtered and concentrated *in vacuo*. The product was purified by an automated flash chromatography system using silica cartridges and the indicated solvent.

Remark recovery thiophenolate leaving group:

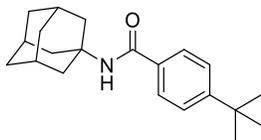
The waste resulting from the thiophenolate leaving group could be recovered as diphenyl disulfide (PhSSPh) by quenching the reaction with an aqueous ammonium hydroxide solution and stirring it for 18 h under oxygen atmosphere. The yield of diphenyl disulfide was determined by the ratio of the mmol of diphenyl disulfide and half of the mmol of the thiocarbamate as two molecules of thiophenolate (substrate) are required to obtain one molecule of diphenyl disulfide.

N-(1-Adamantyl)-2,4,6-triisopropylbenzamide (**3a**) [PMSA1046]



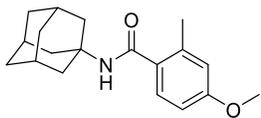
The general procedure B was applied using 1-bromo-2,4,6-triisopropylbenzene (156 mg, 0.55 mmol, 2.2 equiv) and *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 72 mg, 0.25 mmol). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-(1-Adamantyl)-2,4,6-triisopropylbenzamide was obtained in 98% (93 mg) yield. Diphenyl disulfide was isolated in 99% (54 mg) yield. No spectroscopic data have been reported in the literature.

White solid, m.p.: 211-212 °C, $R_f = 0.65$ in heptane / EtOAc (9:1), $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 6.99 (s, 2H), 5.34 (br s, 1H), 3.13 (hept, $J = 6.8$ Hz, 2H), 2.88 (hept, $J = 6.9$ Hz, 1H), 2.12 (s, 9H), 1.87 - 1.65 (m, 6H), 1.27 - 1.24 (m, 18H) ppm. $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 169.7 (C), 149.5 (C), 144.8 (C), 134.9 (C), 121.0 (CH), 52.8 (C), 41.9 (CH_2), 36.5 (CH_2), 34.5 (CH), 30.8 (CH), 29.7 (CH_3), 24.8 (CH), 24.6 (CH), 24.2 (CH_3) ppm. HRMS (ESI) for $\text{C}_{26}\text{H}_{40}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 382.3104, found 382.3109.

***N*-(1-Adamantyl)-4-(*tert*-butyl)benzamide (3b) [PMSA983]**

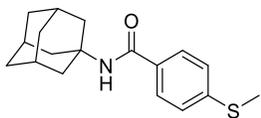
The general procedure B was applied using 4-bromo-*tert*-butylbenzene (95 μ L, 0.55 mmol, 2.2 equiv) and *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 72 mg, 0.25 mmol). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-(1-Adamantyl)-4-(*tert*-butyl)benzamide was obtained in 86% (66 mg) yield. Diphenyl disulfide was isolated in 99% (30 mg) yield. Spectroscopic data have not been reported in the literature.

White solid, m.p.: 188-189 $^{\circ}$ C, R_f = 0.12 in heptane / EtOAc (9:1), ^1H NMR (400 MHz, CDCl_3): δ 7.65 (d, J = 8.3 Hz, 2H), 7.42 (d, J = 8.3 Hz, 2H), 5.76 (br s, 1H), 2.12 (s, 9H), 1.72 (s, 6H), 1.32 (s, 9H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 166.7 (C), 154.6 (C), 133.4 (C), 126.7 (CH), 125.5 (CH), 52.3 (C), 41.9 (CH_2), 36.6 (CH_2), 35.0 (C), 31.3 (CH), 29.7 (CH_3) ppm. HRMS (ESI) for $\text{C}_{21}\text{H}_{30}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 312.2322, found 312.2319.

***N*-(1-Adamantyl)-4-methoxy-2-methylbenzamide (3c) [PMSA970]**

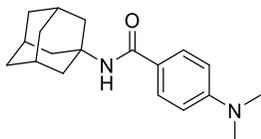
The general procedure B was applied using 1-bromo-4-methoxy-2-methylbenzene (78 μ L, 0.55 mmol, 2.2 equiv) and *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 72 mg, 0.25 mmol). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-(1-Adamantyl)-4-methoxy-2-methylbenzamide was obtained in 85% (64 mg) yield. Diphenyl disulfide was isolated in 88% (27 mg) yield. Spectroscopic data have not been reported in the literature. New compound according to Scifinder.

White solid, m.p.: 170-171 $^{\circ}$ C, R_f = 0.09 in heptane / EtOAc (9:1), ^1H NMR (400 MHz, CDCl_3): δ 7.29 (d, J = 8.3 Hz, 1H), 6.71 - 6.67 (m, 2H), 5.37 (br s, 1H), 3.80 (s, 3H), 2.44 (s, 3H), 2.12 (s, 9H), 1.76 - 1.69 (m, 6H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 169.3 (C), 160.4 (C), 138.1 (C), 130.8 (CH), 128.4 (CH), 116.5 (CH), 110.9 (CH), 55.4 (CH_3), 52.5 (C), 42.0 (CH_2), 36.6 (CH_2), 29.7 (CH), 20.3 (CH_3) ppm. HRMS (ESI) for $\text{C}_{19}\text{H}_{26}\text{NO}_2$ $[\text{M}+\text{H}]^+$ calcd. 300.1958, found 300.1951.

***N*-(1-Adamantyl)-4-(methylsulfanyl)benzamide (3d) [PMSA982]**

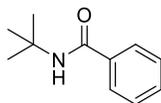
The general procedure B was applied using 4-bromothioanisole (107 mg, 0.53 mmol, 2.2 equiv) and *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 72 mg, 0.25 mmol). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-(1-Adamantyl)-4-(methylsulfanyl)benzamide was obtained in 83% (63 mg) yield. Diphenyl disulfide was isolated in 85% (22 mg) yield. Spectroscopic data have not been reported in the literature. New compound according to Scifinder.

White solid, m.p.: 147-148 $^{\circ}$ C, R_f = 0.12 in heptane / EtOAc (9:1), ^1H NMR (400 MHz, CDCl_3): δ 7.63 (d, J = 8.3 Hz, 2H), 7.24 (d, J = 8.4 Hz, 2H), 5.73 (br s, 1H), 2.50 (s, 3H), 2.12 (s, 9H), 1.76 - 1.69 (m, 6H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 166.2 (C), 142.9 (C), 132.5 (C), 127.3 (CH), 125.7 (CH), 52.4 (C), 41.9 (CH_2), 36.6 (CH_2), 29.7 (CH), 15.4 (CH_3) ppm. HRMS (ESI) for $\text{C}_{18}\text{H}_{24}\text{NOS}$ $[\text{M}+\text{H}]^+$ calcd. 302.1573, found 302.1593.

***N*-(1-Adamantyl)-4-(dimethylamino)benzamide (3e) [PMSA975]**

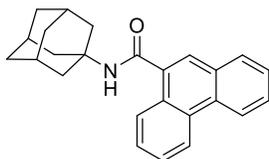
The general procedure B was applied using 4-bromo-*N,N*-dimethylaniline (120 mg, 0.60 mmol, 2.4 equiv) and *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 72 mg, 0.25 mmol). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-(1-Adamantyl)-4-(dimethylamino)benzamide was obtained in 67% (50 mg) yield. Diphenyl disulfide was isolated in 85% (26 mg) yield. Spectroscopic data have not been reported in the literature.

White solid, m.p.: 172-173 $^{\circ}$ C, R_f = 0.11 in heptane / EtOAc (9:1), ^1H NMR (400 MHz, CDCl_3): δ 7.62 (d, J = 8.9 Hz, 2H), 6.65 (d, J = 8.9 Hz, 2H), 5.67 (br s, 1H), 3.00 (s, 6H), 2.12 (s, 9H), 1.75 - 1.68 (m, 6H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 166.8 (C), 152.4 (C), 128.2 (CH_2), 123.1 (C), 111.2 (CH_2), 52.0 (C), 42.1 (CH_2), 40.3 (CH), 36.7 (CH_2), 29.7 (CH_3) ppm. HRMS (ESI) for $\text{C}_{19}\text{H}_{27}\text{N}_2\text{O}$ $[\text{M}+\text{H}]^+$ calcd. 299.2118, found 299.2133.

***N*-tert-Butylbenzamide (3f) [PMSA827b/864]**

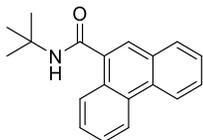
The general procedure A was applied using *S*-phenyl *tert*-butylthiocarbamate (**1b**, 105 mg, 0.5 mmol) and phenylmagnesium bromide (**1g**, 367 μ L, 3.0 M solution in tetrahydrofuran, 1.1 mmol, 2.2 equiv). The mixture was stirred for 4 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 40% ethyl acetate in 25 min, 25 mL/min). *N*-*tert*-Butylbenzamide was obtained in 91% (80 mg) yield. Diphenyl disulfide was isolated in 89% (48 mg) yield. The spectroscopic data are in accordance with the literature.¹²

White solid, m.p.: 134-135 °C (lit.: 135 °C),¹³ R_f = 0.15 in heptane / EtOAc (9:1), ¹H NMR (400 MHz, CDCl₃): δ 7.72 - 7.70 (m, 2H), 7.48 - 7.44 (m, 1H), 7.42 - 7.38 (m, 2H), 5.93 (br s, 1H), 1.47 (s, 9H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 167.0 (C), 136.1 (C), 131.2 (CH), 128.6 (CH), 126.8 (CH), 51.7 (C), 29.0 (CH₃) ppm. HRMS (ESI) for C₁₁H₁₆NO [M+H]⁺ calcd. 178.1226, found 178.1234.

***N*-(1-Adamantyl)phenanthrene-9-carboxamide (3g) [PMSA972]**

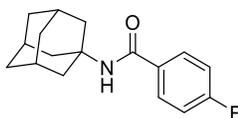
The general procedure B was applied using 9-bromophenanthrene (154 mg, 0.60 mmol, 2.4 equiv) and *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 72 mg, 0.25 mmol). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 20% ethyl acetate in 25 min, 25 mL/min). *N*-(1-Adamantyl)phenanthrene-9-carboxamide was obtained in 72% (64 mg) yield. Diphenyl disulfide was isolated in 81% (22 mg) yield. Spectroscopic data have not been reported in the literature. New compound according to Scifinder.

White solid, m.p.: 224-225 °C, R_f = 0.25 in heptane / EtOAc (9:1), ¹H NMR (400 MHz, CDCl₃): δ 8.69 (dd, J = 14.2, 8.2 Hz, 2H), 8.32 (d, J = 7.9 Hz, 1H), 7.89 (d, J = 7.8 Hz, 1H), 7.81 (s, 1H), 7.71 - 7.62 (m, 4H), 5.75 (br s, 1H), 2.24 (s, 6H), 2.18 (br s, 3H), 1.81 - 1.74 (m, 6H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 169.1 (C), 135.2 (C), 131.0 (C), 130.8 (C), 130.7 (C), 129.1 (CH), 128.9 (C), 127.8 (CH), 127.3 (CH), 127.2 (CH), 127.2 (CH), 126.4 (CH), 125.6 (CH), 123.0 (CH), 122.8 (CH), 53.0 (C), 42.0 (CH₂), 36.6 (CH₂), 29.7 (CH) ppm. HRMS (ESI) for C₂₅H₂₆NO [M+H]⁺ calcd. 356.2009, found 356.2002.

***N*-tert-Butylphenanthrene-9-carboxamide (3h) [PMSA973]**

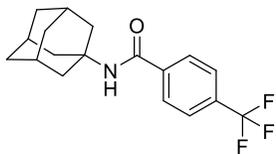
The general procedure B was applied using 9-bromophenanthrene (154 mg, 0.60 mmol, 2.4 equiv) and *S*-phenyl *tert*-butylthiocarbamate (**1b**, 52 mg, 0.25 mmol). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 20% ethyl acetate in 25 min, 25 mL/min). *N*-*tert*-Butylphenanthrene-9-carboxamide was obtained in 76% (53 mg) yield. Diphenyl disulfide was isolated in 99% (28 mg) yield. The spectroscopic data are in accordance with the literature.¹⁴

White solid, m.p.: 155-156 °C, R_f = 0.21 in heptane / EtOAc (9:1), ¹H NMR (400 MHz, CDCl₃): δ 8.69 (dd, J = 13.8, 8.2 Hz, 2H), 8.30 (d, J = 7.9 Hz, 1H), 7.89 (d, J = 7.8 Hz, 1H), 7.80 (s, 1H), 7.71 - 7.62 (m, 4H), 5.91 (br s, 1H), 1.58 (s, 9H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 169.3 (C), 135.1 (C), 131.0 (C), 130.8 (C), 130.7 (C), 129.1 (CH), 128.9 (C), 127.8 (CH), 127.3 (CH), 127.2 (CH), 127.1 (CH), 126.4 (CH), 125.6 (CH), 123.0 (CH), 122.8 (CH), 52.3 (C), 29.1 (CH₃) ppm. HRMS (ESI) for C₁₉H₂₀NO [M+H]⁺ calcd. 278.1539, found 278.1549.

***N*-(1-Adamantyl)-4-fluorobenzamide (3i) [PMSA976/1048]**

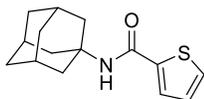
The general procedure B was applied using 1-bromo-4-fluorobenzene (121 μ L, 1.1 mmol, 2.2 equiv) and *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 144 mg, 0.25 mmol) and Mg turnings (27 mg, 1.1 mmol, 1.0 equiv). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-(1-Adamantyl)-4-fluorobenzamide was obtained in 95% (130 mg) yield. Diphenyl disulfide was isolated in 90% (49 mg) yield. Spectroscopic data have not been reported in the literature.

White solid, m.p.: 161-162 °C, R_f = 0.26 in heptane / EtOAc (9:1), ¹H NMR (400 MHz, CDCl₃): δ 7.72 (dd, J = 8.6, 5.4 Hz, 2H), 7.08 (m, 2H), 5.70 (br s, 1H), 2.12 (s, 9H), 1.74 - 1.71 (m, 6H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 165.7 (C), 164.5 (d, J_{C-F} = 233.2 Hz, C), 132.4 (d, J_{C-F} = 3.3 Hz, C), 129.1 (d, J_{C-F} = 8.9 Hz, CH), 115.6 (d, J_{C-F} = 21.8 Hz, CH), 52.6 (C), 41.9 (CH₂), 36.6 (CH₂), 29.7 (CH) ppm. HRMS (ESI) for C₁₇H₂₁FNO [M+H]⁺ calcd. 274.1602, found 274.1615.

***N*-(1-Adamantyl)-4-(trifluoromethyl)benzamide (3j) [PMSA989]**

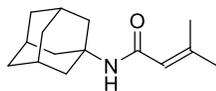
The general procedure B was applied using 4-bromobenzotrifluoride (77 μ L, 0.55 mmol, 2.2 equiv) and *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 72 mg, 0.25 mmol). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 20% ethyl acetate in 25 min, 25 mL/min). *N*-(1-Adamantyl)-4-(trifluoromethyl)benzamide was obtained in 84% (68 mg) yield. Diphenyl disulfide was isolated in 82% (22 mg) yield. Spectroscopic data have not been reported in the literature.

White solid, m.p.: 160-161 $^{\circ}$ C, R_f = 0.67 in heptane / EtOAc (2:1), ^1H NMR (400 MHz, CDCl_3): δ 7.81 (d, J = 8.1 Hz, 2H), 7.67 (d, J = 8.1 Hz, 2H), 5.78 (br s, 1H), 2.13 (s, 9H), 1.74 (s, 6H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 165.5 (C), 139.6 (C), 133.0 (q, $J_{\text{C-F}}$ = 32.8 Hz, C), 127.4 (CH), 125.7 (q, $J_{\text{C-F}}$ = 3.7 Hz, CH), 123.9 (q, $J_{\text{C-F}}$ = 272.4 Hz, C), 52.9 (C), 41.8 (CH_2), 36.5 (CH_2), 29.7 (CH) ppm. HRMS (ESI) for $\text{C}_{18}\text{H}_{21}\text{F}_3\text{NO}$ [$\text{M}+\text{H}$] $^+$ calcd. 324.1570, found 324.1588.

***N*-(1-Adamantyl)thiophene-2-carboxamide (3k) [PMSA974]**

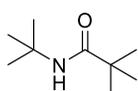
The general procedure B was applied using 2-bromothiophene (58 μ L, 0.60 mmol, 2.4 equiv) and *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 72 mg, 0.25 mmol). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-(1-Adamantyl)thiophene-2-carboxamide was obtained in 82% (53 mg) yield. Diphenyl disulfide was isolated in 81% (24 mg) yield. Spectroscopic data have not been reported in the literature.

White solid, m.p.: 197-198 $^{\circ}$ C, R_f = 0.15 in heptane / EtOAc (9:1), ^1H NMR (400 MHz, CDCl_3): δ 7.42 - 7.40 (m, 2H), 7.04 (dd, J = 4.9, 3.8 Hz, 1H), 5.64 (br s, 1H), 2.12 (s, 9H), 1.72 (s, 6H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 161.1 (C), 140.9 (C), 129.4 (CH), 127.6 (CH), 127.5 (CH), 52.8 (C), 41.9 (CH_2), 36.5 (CH_2), 29.6 (CH) ppm. HRMS (ESI) for $\text{C}_{15}\text{H}_{20}\text{NOS}$ [$\text{M}+\text{H}$] $^+$ calcd. 262.1260, found 262.1278.

***N*-(1-Adamantyl)-3-methylbut-2-enamide (3l) [PMSA949]**

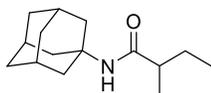
The general procedure A was applied using *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 144 mg, 0.5 mmol) and 2-methyl-1-propenylmagnesium bromide (2.2 mL, 0.5 M solution in tetrahydrofuran, 1.1 mmol, 2.2 equiv). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-(1-Adamantyl)-3-methylbut-2-enamide was obtained in 82% (95 mg) yield. Diphenyl disulfide was isolated in 90% (49 mg) yield. Spectroscopic data have not been reported in the literature.

White solid, m.p.: 171-172 $^{\circ}$ C, R_f = 0.34 in heptane / EtOAc (9:1), ^1H NMR (400 MHz, CDCl_3): δ 5.48 (s, 1H), 5.02 (br s, 1H), 2.11 - 2.03 (m, 12H), 1.80 (s, 3H), 1.73 - 1.64 (m, 6H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 166.6 (C), 149.5 (C), 120.0 (CH), 51.9 (C), 42.0 (CH_2), 36.6 (CH_2), 29.7 (CH_3), 27.2 (CH), 19.6 (CH) ppm. HRMS (ESI) for $\text{C}_{15}\text{H}_{24}\text{NO}$ [$\text{M}+\text{H}$] $^+$ calcd. 234.1852, found 234.1851.

***N*-tert-Butylpivalamide (3m) [PMSA945]**

The general procedure B was applied using *S*-phenyl *tert*-butylthiocarbamate (**1b**, 105 mg, 0.5 mmol) and *tert*-butylbromide (150 mg, 1.1 mmol, 2.2 equiv). The mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 30% ethyl acetate in 25 min, 25 mL/min). *N*-*tert*-Butylpivalamide was obtained in 81% (80 mg) yield. Diphenyl disulfide was isolated in 76% (41 mg) yield. The spectroscopic data are in accordance with the literature.¹⁵

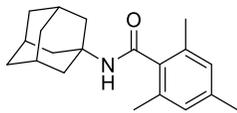
White solid, m.p.: 61-62 $^{\circ}$ C, R_f = 0.36 in heptane / EtOAc (9:1), ^1H NMR (400 MHz, CDCl_3): δ 5.39 (br s, 1H), 1.34 (s, 9H), 1.16 (s, 9H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 178.0 (C), 50.8 (C), 39.1 (C), 28.9 (CH_3), 27.8 (CH_3) ppm. HRMS (ESI) for $\text{C}_9\text{H}_{20}\text{NO}$ [$\text{M}+\text{H}$] $^+$ calcd. 158.2645, found 158.2649.

***N*-(1-Adamantyl)-2-methylbutanamide (3n) [PMSA1043]**

The general procedure A was applied using *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 144 mg, 0.5 mmol) and *sec*-butylmagnesium bromide (1.1 mL, 1.0 M solution in tetrahydrofuran, 1.1 mmol, 2.2 equiv). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-(1-Adamantyl)-2-methylbutanamide was obtained in 87% (102 mg) yield. Diphenyl disulfide was isolated in 92% (51 mg) yield. Spectroscopic data have not been reported in the literature.

White solid, m.p.: 151-152 °C, $R_f = 0.22$ in heptane / EtOAc (9:1), $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 5.07 (br s, 1H), 2.06 (br s, 3H), 2.00 - 1.92 (m, 7H), 1.70 - 1.57 (m, 7H), 1.43 - 1.32 (m, 1H), 1.08 (d, $J = 6.8$ Hz, 3H), 0.89 (t, $J = 7.4$ Hz, 3H) ppm. $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 175.8 (C), 51.8 (C), 44.2 (CH), 41.9 (CH_2), 36.6 (CH_2), 29.6 (CH_3), 27.7 (CH_2), 17.8 (CH), 12.1 (CH_3) ppm. HRMS (ESI) for $\text{C}_{15}\text{H}_{26}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 236.2009, found 236.2014.

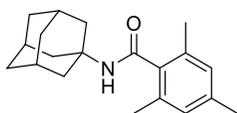
N-(1-Adamantyl)-2,4,6-trimethylbenzamide (4a) [PMSA873/PMSA-CVE-017]



The general procedure A was applied using *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 144 mg, 0.5 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 1.1 mL, 1.0 M solution in tetrahydrofuran, 1.1 mmol, 2.2 equiv). The mixture was stirred for 4 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 20 min, 25 mL/min). *N*-(1-Adamantyl)-2,4,6-trimethylbenzamide was obtained in 93% (137 mg) yield. Diphenyl disulfide was isolated in 93% (51 mg) yield. The spectroscopic data are in accordance with the literature.⁶

White solid, m.p.: 228-229 °C (lit.: 225 °C),⁶ $R_f = 0.69$ in heptane / EtOAc (9:1), $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 6.82 (s, 2H), 5.28 (br s, 1H), 2.31 (s, 6H), 2.26 (s, 3H), 2.12 (s, 9H), 1.76 - 1.69 (m, 6H) ppm. $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 169.6 (C), 138.1 (C), 136.3 (C), 134.0 (C), 128.2 (CH), 52.6 (C), 42.0 (CH_2), 36.6 (CH_2), 29.7 (CH_3), 21.2 (CH), 19.1 (CH_3) ppm. HRMS (ESI) for $\text{C}_{20}\text{H}_{28}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 298.2165, found 298.2180.

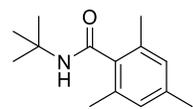
Large scale preparation of *N*-(1-Adamantyl)-2,4,6-trimethylbenzamide (4a) [PMSA1037]



A Schlenk flask of 100 mL was flame-dried under vacuum and cooled to room temperature under an argon flow. Subsequently, *S*-phenyl (1-adamantyl)thiocarbamate (**1a**, 1.437 g, 5.0 mmol) and 2-methyltetrahydrofuran (25 mL, dry) were added and the solution was cooled to 0 °C. Subsequently 2,4,6-trimethylphenylmagnesium bromide (**2o**, 11.0 mL, 1.0 M solution in tetrahydrofuran, 11.0 mmol, 2.2 equiv) was added dropwise over 10 minutes under argon atmosphere and slowly warmed to room temperature.

After being stirred for 2 h, the mixture was quenched with NH_4OH (20 mL). The vial was flushed with oxygen and stirred for 18 h under oxygen atmosphere (balloon). The 2-methyltetrahydrofuran was evaporated, water (10 mL) was added and the mixture was extracted with ethyl acetate (3 x 20 mL). The combined organic phases were dried over anhydrous MgSO_4 , filtered and concentrated *in vacuo*. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 30% ethyl acetate in 20 min, 25 mL/min). *N*-(1-Adamantyl)-2,4,6-trimethylbenzamide was obtained in 92% (1.373 g) yield. Diphenyl disulfide was isolated in 90% (491 mg) yield.

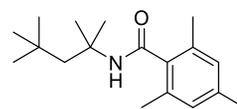
N-(*tert*-Butyl)-2,4,6-trimethylbenzamide (4b) [PMSA987]



The general procedure A was applied using *S*-phenyl *tert*-butylthiocarbamate (**1b**, 52 mg, 0.25 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 550 μL , 1.0 M solution in tetrahydrofuran, 0.55 mmol, 2.2 equiv). The mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 20 min, 25 mL/min). *N*-(*tert*-butyl)-2,4,6-trimethylbenzamide was obtained in 92% (50 mg) yield. Diphenyl disulfide was isolated in 91% (25 mg) yield. The spectroscopic data are in accordance with the literature.⁶

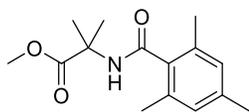
White solid, m.p.: 142-143 °C (lit.: 143 °C),⁶ $R_f = 0.24$ in heptane / EtOAc (9:1), $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 6.82 (s, 2H), 5.42 (br s, 1H), 2.30 (s, 6H), 2.26 (s, 3H), 1.45 (s, 9H) ppm. $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 169.9 (C), 138.2 (C), 136.2 (C), 134.1 (C), 128.3 (CH), 51.8 (C), 29.0 (CH_3), 21.2 (CH_3), 19.0 (CH_3) ppm. HRMS (ESI) for $\text{C}_{14}\text{H}_{22}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 220.1696, found 220.1710.

2,4,6-Trimethyl-*N*-(2,4,4-trimethylpentan-2-yl)benzamide (4c) [PMSA986/PMS-CVE-035]



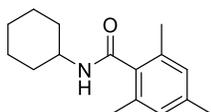
The general procedure A was applied using *S*-phenyl (2,4,4-trimethylpentan-2-yl)thiocarbamate (**1c**, 66 mg, 0.25 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 550 μL , 1.0 M solution in tetrahydrofuran, 0.55 mmol, 2.2 equiv). The mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). 2,4,6-Trimethyl-*N*-(2,4,4-trimethylpentan-2-yl)benzamide was obtained in 94% (65 mg) yield. Diphenyl disulfide was isolated in 91% (25 mg) yield. The spectroscopic data are in accordance with the literature.⁶

White solid, m.p.: 123-124 °C (lit.: 125 °C),⁶ $R_f = 0.27$ in heptane / EtOAc (9:1), $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 6.82 (s, 2H), 5.43 (br s, 1H), 2.32 (s, 6H), 2.26 (s, 3H), 1.75 (s, 2H), 1.57 (s, 6H), 1.04 (s, 9H) ppm. $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 169.6 (C), 138.1 (C), 136.3 (C), 134.1 (C), 128.4 (CH), 56.3 (C), 54.2 (CH_2), 31.8 (CH_3), 31.8 (C), 28.5 (CH_3), 21.2 (CH_3), 19.2 (CH_3) ppm. HRMS (ESI) for $\text{C}_{18}\text{H}_{30}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 276.2322, found 276.2337.

Methyl 2-methyl-N-(2,4,6-trimethylbenzoyl)alaninate (4d) [PMSA1034]


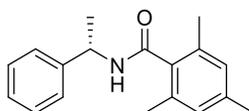
The general procedure A was applied using methyl 2-methyl-2-[(phenylthio)carbonyl]amino}propanoate (**1d**, 63 mg, 0.25 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 550 μ L, 1.0 M solution in tetrahydrofuran, 0.55 mmol, 2.2 equiv). The mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). Methyl 2-methyl-N-(2,4,6-trimethylbenzoyl)alaninate was obtained in 83% (55 mg) yield. Diphenyl disulfide was isolated in 84% (23 mg) yield. The spectroscopic data are in accordance with the literature.⁶

White oil, R_f = 0.41 in heptane / EtOAc (2:1), ¹H NMR (400 MHz, CDCl₃): δ 6.81 (s, 2H), 6.19 (br s, 1H), 3.77 (s, 3H), 2.29 (s, 6H), 2.26 (s, 3H), 1.63 (s, 6H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 174.9 (C), 169.8 (C), 138.5 (C), 134.6 (C), 134.5 (C), 128.3 (CH), 56.7 (C), 52.6 (CH₃), 24.9 (CH₃), 21.2 (CH₃), 18.9 (CH₃) ppm. HRMS (ESI) for C₁₅H₂₂NO₃ [M+H]⁺ calcd. 264.1594, found 264.1599.

N-Cyclohexyl-2,4,6-trimethylphenylcarboxamide (4e) [PMS-CVE-032]


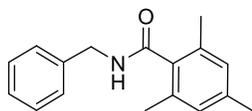
The general procedure A was applied using S-phenyl cyclohexylthiocarbamate (**1e**, 59 mg, 0.25 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 550 μ L, 1.0 M solution in tetrahydrofuran, 0.55 mmol, 2.2 equiv). The mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). N-Cyclohexyl-2,4,6-trimethylphenylcarboxamide was obtained in 86% (53 mg) yield. Diphenyl disulfide was isolated in 82% (20 mg) yield. The spectroscopic data are in accordance with the literature.¹⁶

White solid, m.p.: 168-170 °C (lit.: 171-172 °C),¹⁶ R_f = 0.71 in heptane / EtOAc (9:1), ¹H NMR (400 MHz, CDCl₃): δ 6.83 (s, 2H), 5.50 (d, J = 6.5 Hz, 1H), 4.06 - 3.99 (m, 1H), 2.29 (s, 6H), 2.26 (s, 3H), 2.06 - 2.03 (m, 2H), 1.75 - 1.71 (m, 2H), 1.66 - 1.63 (m, 1H), 1.45 - 1.42 (m, 2H), 1.25 - 1.18 (m, 3H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 169.6 (C), 138.4 (C), 135.4 (C), 134.2 (C), 128.2 (CH), 48.3 (CH), 33.4 (CH₂), 25.7 (CH₂), 25.0 (CH₂), 21.2 (CH₃), 19.1 (CH₃) ppm. HRMS (ESI) for C₁₆H₂₄NO [M+H]⁺ calcd. 246.1852, found 246.1851.

(S)-2,4,6-Trimethyl-N-(1-phenylethyl)benzamide (4f) [PMSA1015]


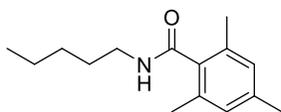
The general procedure A was applied using S-phenyl (1-phenylethyl)thiocarbamate (**1f**, 64 mg, 0.25 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 550 μ L, 1.0 M solution in tetrahydrofuran, 0.55 mmol, 2.2 equiv). The mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 25% ethyl acetate in 25 min, 25 mL/min). (S)-2,4,6-Trimethyl-N-(1-phenylethyl)benzamide was obtained in 84% (56 mg) yield. Diphenyl disulfide was isolated in 85% (23 mg) yield. Spectroscopic data have not been reported in the literature. New compound according to Scifinder.

White solid, m.p.: 139-140 °C, R_f = 0.46 in heptane / EtOAc (2:1), ¹H NMR (400 MHz, CDCl₃): δ 7.39 - 7.33 (m, 4H), 7.29 - 7.26 (m, 1H), 6.82 (s, 2H), 5.84 (d, J = 7.1 Hz, 1H), 5.42 - 5.35 (m, 1H), 2.25 (s, 9H), 1.60 (d, J = 6.8 Hz, 3H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 169.6 (C), 143.0 (C), 138.6 (C), 135.0 (C), 134.3 (C), 128.9 (CH), 128.3 (CH), 127.6 (CH), 126.5 (CH), 49.0 (CH), 21.7 (CH₃), 21.2 (CH₃), 19.2 (CH₃) ppm. HRMS (ESI) for C₁₈H₂₂NO [M+H]⁺ calcd. 268.1696, found 268.1692.

N-Benzyl-2,4,6-trimethylbenzamide (4g) [PMSA1014]


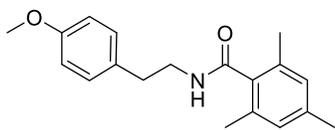
The general procedure A was applied using S-phenyl benzylthiocarbamate (**1g**, 61 mg, 0.25 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 550 μ L, 1.0 M solution in tetrahydrofuran, 0.55 mmol, 2.2 equiv). The mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 50% ethyl acetate in 25 min, 25 mL/min). N-Benzyl-2,4,6-trimethylbenzamide was obtained in 79% (50 mg) yield. Diphenyl disulfide was isolated in 88% (24 mg) yield. Spectroscopic data have not been reported in the literature.

White solid, m.p. 145-146 °C, R_f = 0.07 in heptane / EtOAc (9:1), ¹H NMR (400 MHz, CDCl₃): δ 7.36 - 7.27 (m, 5H), 6.82 (s, 2H), 5.87 (br s, 1H), 4.63 (d, J = 5.7 Hz, 2H), 2.28 (s, 6H), 2.26 (s, 3H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 170.5 (C), 138.6 (C), 138.2 (C), 134.9 (C), 134.3 (C), 128.9 (CH), 128.4 (CH), 128.2 (CH), 127.8 (CH), 44.0 (CH), 21.2 (CH₃), 19.3 (CH₃) ppm. HRMS (ESI) for C₁₇H₂₀NO [M+H]⁺ calcd. 254.1539, found 254.1548.

2,4,6-Trimethyl-N-pentylbenzamide (4h) [PMSA1016]

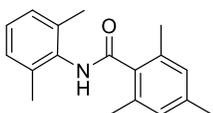
The general procedure A was applied using *S*-phenyl pentylthiocarbamate (**1h**, 56 mg, 0.25 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 550 μ L, 1.0 M solution in tetrahydrofuran, 0.55 mmol, 2.2 equiv). The mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 50% ethyl acetate in 25 min, 25 mL/min). 2,4,6-Trimethyl-*N*-pentylbenzamide was obtained in 83% (48 mg) yield. Diphenyl disulfide was isolated in 82% (22 mg) yield. Spectroscopic data have not been reported in the literature. New compound according to Scifinder.

Off-white solid, m.p.: 72-73 °C, R_f = 0.49 in heptane / EtOAc (2:1), ^1H NMR (400 MHz, CDCl_3): δ 6.82 (s, 2H), 5.62 (br s, 1H), 3.43 (q, J = 6.8 Hz, 2H), 2.28 (s, 6H), 2.26 (s, 3H), 1.61 - 1.58 (m, 2H), 1.37 - 1.30 (m, 4H), 0.91 (t, J = 6.7 Hz, 3H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 170.6 (C), 138.4 (C), 135.4 (C), 134.3 (C), 128.3 (CH), 39.7 (CH_2), 29.5 (CH_2), 29.3 (CH_2), 22.4 (CH_2), 21.2 (CH_3), 19.2 (CH_3), 14.1 (CH_3) ppm. HRMS (ESI) for $\text{C}_{15}\text{H}_{24}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 234.1852, found 234.1847.

***N*-(4-Methoxyphenylethyl)-2,4,6-trimethylbenzamide (4i) [PMS-CVE-018]**

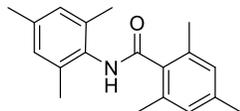
The general procedure A was applied using *S*-phenyl 4-methoxyphenylethylthiocarbamate (**1i**, 72 mg, 0.25 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 550 μ L, 1.0 M solution in tetrahydrofuran, 0.55 mmol, 2.2 equiv). The mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 50% ethyl acetate in 35 min, 25 mL/min). *N*-(4-methoxyphenylethyl)-2,4,6-trimethylbenzamide was obtained in 75% (58 mg) yield. Diphenyl disulfide was isolated in 72% (16 mg) yield. Spectroscopic data have not been reported in the literature.

White solid, m.p.: 148-149 °C, R_f = 0.10 in heptane / EtOAc (9:1), ^1H NMR (400 MHz, CDCl_3): δ 7.14 (d, J = 8.4 Hz, 2H), 6.84 (d, J = 8.5 Hz, 2H), 6.80 (s, 2H), 5.58 (br s, 1H), 3.78 (s, 3H), 3.73 (dd, J = 12.9, 6.7 Hz, 2H), 2.87 (t, J = 6.9 Hz, 2H), 2.25 (s, 3H), 2.22 (s, 6H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 170.6 (C), 158.5 (C), 138.5 (C), 135.2 (C), 134.3 (C), 130.7 (C), 129.8 (CH), 128.3 (CH), 114.3 (CH), 55.4 (CH_3), 40.7 (CH_2), 35.0 (CH_2), 21.2 (CH_3), 19.2 (CH_3) ppm. HRMS (ESI) for $\text{C}_{19}\text{H}_{24}\text{NO}_2$ $[\text{M}+\text{H}]^+$ calcd. 298.1802, found 298.1803.

***N*-(2,6-Dimethylphenyl)-2,4,6-trimethylbenzamide (4j) [PMSA988]**

The general procedure A was applied using *S*-phenyl 2,6-dimethylphenylthiocarbamate (**1j**, 64 mg, 0.25 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 550 μ L, 1.0 M solution in tetrahydrofuran, 0.55 mmol, 2.2 equiv). The mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-(2,6-Dimethylphenyl)-2,4,6-trimethylbenzamide was obtained in 73% (48 mg) yield. Diphenyl disulfide was isolated in 85% (23 mg) yield. The spectroscopic data are in accordance with the literature.²

White solid, m.p.: 195-196 °C (lit.: 197-199 °C),² R_f = 0.16 in heptane / EtOAc (9:1), ^1H NMR (400 MHz, CDCl_3): δ 7.13 (s, 3H), 6.92 - 6.90 (m, 3H), 2.48 (s, 6H), 2.39 (s, 6H), 2.31 (s, 3H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 168.8 (C), 138.9 (C), 135.3 (C), 135.1 (C), 134.7 (C), 133.7 (C), 128.8 (CH), 128.8 (CH), 127.6 (CH), 21.2 (CH_3), 20.1 (CH_3), 19.7 (CH_3) ppm. HRMS (ESI) for $\text{C}_{18}\text{H}_{22}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 268.1696, found 268.1695.

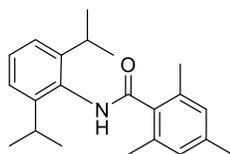
***N*-(2,4,6-Trimethylphenyl)-2,4,6-trimethylbenzamide (4k) [PMSA1027]**

The general procedure A was applied using *S*-phenyl 2,4,6-trimethylphenylthiocarbamate (**1k**, 68 mg, 0.25 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 550 μ L, 1.0 M solution in tetrahydrofuran, 0.55 mmol, 2.2 equiv). The reaction mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 40% ethyl acetate in 30 min, 25 mL/min). *N*-(2,4,6-Trimethylphenyl)-2,4,6-trimethylbenzamide was obtained in 92% (65 mg) yield. Diphenyl disulfide was isolated in 95% (26 mg) yield. The spectroscopic data are in accordance with the literature.²

White solid, m.p.: 206-207 °C (lit.: 205-206 °C),² R_f = 0.11 in heptane / EtOAc (9:1), ^1H NMR (400 MHz, CDCl_3): δ 6.94 (s, 2H), 6.90 (s, 2H), 6.87 (br s, 1H), 2.47 (s, 6H), 2.35 (s, 6H), 2.31 (s, 3H), 2.29 (s, 3H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 168.9 (C), 138.8 (C), 137.3 (C), 135.1 (C), 135.1 (C), 134.7

(C), 131.1 (C), 129.5 (CH), 128.7 (CH), 21.2 (CH₃), 21.0 (CH₃), 20.1 (CH₃), 19.5 (CH₃) ppm. HRMS (ESI) for C₁₉H₂₄NO [M+H]⁺ calcd. 282.1852, found 282.1849.

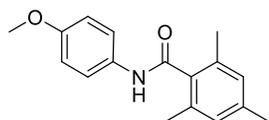
N-(2,6-Diisopropylphenyl)-2,4,6-trimethylbenzamide (**4l**) [PMS-CVE-033/PMS-CVE-045]



The general procedure A was applied using *S*-phenyl (2,6-diisopropylphenyl)thiocarbamate (**1l**, 78 mg, 0.25 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 550 μ L, 1.0 M solution in tetrahydrofuran, 0.55 mmol, 2.2 equiv). The mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-(2,6-Diisopropylphenyl)-2,4,6-trimethylbenzamide was obtained in 67% (57 mg) yield. Diphenyl disulfide was isolated in 50% (10 mg) yield. The spectroscopic data are in accordance with the literature.⁶

White solid, m.p.: 215-216 °C (lit.: 217 °C),⁶ R_f = 0.74 in heptane / EtOAc (9:1), ¹H NMR (400 MHz, CDCl₃): δ 7.35 - 7.31 (m, 1H), 7.24 - 7.22 (m, 2H), 6.92 (s, 2H), 6.86 (s, 1H), 3.36 (hp, J = 6.9 Hz, 2H), 2.49 (s, 6H), 2.32 (s, 3H), 1.26 (d, J = 6.8 Hz, 12H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 170.0 (C), 146.3 (C), 139.0 (C), 135.0 (C), 134.9 (C), 130.6 (C), 129.0 (CH), 128.6 (CH), 123.9 (CH), 28.9 (CH₃), 24.1 (CH₃), 21.2 (CH), 20.1 (CH₃) ppm. HRMS (ESI) for C₂₂H₃₀NO [M+H]⁺ calcd. 324.2322, found 324.2335.

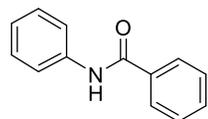
N-(4-Methoxyphenyl)-2,4,6-trimethylbenzamide (**4m**) [PMSA992]



The general procedure A was applied using *S*-phenyl (4-methoxyphenyl)thiocarbamate (**1m**, 65 mg, 0.25 mmol) and 2,4,6-trimethylphenylmagnesium bromide (**2o**, 550 μ L, 1.0 M solution in tetrahydrofuran, 0.55 mmol, 2.2 equiv). The mixture was stirred for 2 h. The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-(4-Methoxyphenyl)-2,4,6-trimethylbenzamide was obtained in 82% (55 mg) yield. Diphenyl disulfide was isolated in 79% (22 mg) yield. Spectroscopic data have not been reported in the literature.

Off-white solid, m.p.: 202-204 °C, R_f = 0.06 in heptane / EtOAc (9:1), ¹H NMR (400 MHz, CDCl₃): δ 7.52 (d, J = 8.9 Hz, 2H), 7.17 (br s, 1H), 6.91 (d, J = 8.9 Hz, 2H), 6.88 (s, 2H), 3.81 (s, 3H), 2.36 (s, 6H), 2.30 (s, 3H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 168.7 (C), 156.9 (C), 139.0 (C), 135.2 (C), 134.5 (C), 131.1 (C), 128.5 (CH), 121.9 (CH), 114.5 (CH), 55.7 (CH₃), 21.3 (CH₃), 19.3 (CH₃) ppm. HRMS (ESI) for C₁₇H₂₀NO₂ [M+H]⁺ calcd. 270.1489, found 270.1485.

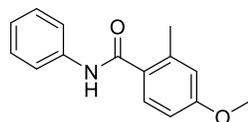
N-Phenylbenzamide (**6a**) [PMSA953]



The general procedure A was applied using *N,S*-diphenylthiocarbamate (**8**, 115 mg, 0.5 mmol) and phenylmagnesium bromide (**2f**, 1.1 mL, 1.0 M solution in tetrahydrofuran, 1.1 mmol, 2.2 equiv). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-Phenylbenzamide was obtained in 24% (24 mg) yield. Diphenyl disulfide was isolated in 90% (100 mg) yield. The spectroscopic data are in accordance with the literature.¹⁷

White solid, m.p.: 157-158 °C (lit.: 159-160 °C),¹⁸ R_f = 0.12 in heptane / EtOAc (9:1), ¹H NMR (400 MHz, CDCl₃): δ 7.87 (d, J = 7.2 Hz, 2H), 7.84 (br s, 1H), 7.65 (d, J = 7.8 Hz, 2H), 7.55 (t, J = 7.3 Hz, 1H), 7.48 (t, J = 7.4 Hz, 2H), 7.37 (t, J = 7.9 Hz, 2H), 7.16 (t, J = 7.4 Hz, 1H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 165.9 (C), 138.1 (C), 135.2 (C), 132.0 (CH), 129.4 (CH), 128.9 (CH), 127.2 (CH), 124.7 (CH), 120.4 (CH) ppm. HRMS (ESI) for C₁₃H₁₁NONa [M+Na]⁺ calcd. 220.0738, found 220.0739.

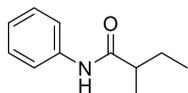
4-Methoxy-2-methyl-*N*-phenylbenzamide (**6b**) [PMSA971]



The general procedure B was applied using 1-bromo-4-methoxy-2-methylbenzene (85 μ L, 0.60 mmol, 2.4 equiv) and *N,S*-diphenylthiocarbamate (**8**, 57 mg, 0.25 mmol). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 20% ethyl acetate in 25 min, 25 mL/min). 4-Methoxy-2-methyl-*N*-phenylbenzamide was obtained in 90% (54 mg) yield. Diphenyl disulfide was isolated in 88% (26 mg) yield. Spectroscopic data have not been reported in the literature.

White solid, m.p.: 182-183 °C, $R_f = 0.09$ in heptane / EtOAc (9:1), $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.59 (d, $J = 7.6$ Hz, 2H), 7.46 (d, $J = 8.6$ Hz, 2H), 7.36 (t, $J = 7.5$ Hz, 2H), 7.14 (t, $J = 7.4$ Hz, 1H), 6.78 - 6.75 (m, 2H), 3.83 (s, 3H), 2.51 (s, 3H) ppm. $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 167.9 (C), 161.1 (C), 139.3 (C), 138.4 (C), 129.2 (CH), 129.0 (C), 128.6 (CH), 124.5 (CH), 120.0 (CH), 117.0 (CH), 111.2 (CH), 55.5 (CH_3), 20.5 (CH_3) ppm. HRMS (ESI) for $\text{C}_{15}\text{H}_{16}\text{NO}_2$ $[\text{M}+\text{H}]^+$ calcd. 242.1176, found 242.1172.

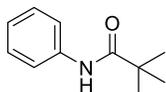
2-Methyl-*N*-phenylbutanamide (6c) [PMSA956]



The general procedure A was applied using *N,S*-diphenylthiocarbamate (**8**, 115 mg, 0.5 mmol) and *sec*-butyllithium (**5c**, 786 μL , 1.4 M solution in tetrahydrofuran, 1.1 mmol, 2.2 equiv). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). 2-Methyl-*N*-phenylbutanamide was obtained in 68% (59 mg) yield. Diphenyl disulfide was isolated in 72% (39 mg) yield. Spectroscopic data are in accordance with the literature.¹⁹

White solid, m.p.: 109-110 °C (lit.: 104 °C),²⁰ $R_f = 0.12$ in heptane / EtOAc (9:1), $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.53 (d, $J = 7.9$ Hz, 2H), 7.32 (t, $J = 7.9$ Hz, 2H), 7.12 - 7.08 (m, 2H), 2.30 - 2.29 (m, 1H), 1.84 - 1.72 (m, 1H), 1.56 - 1.46 (m, 1H), 1.24 (d, $J = 6.8$ Hz, 3H), 0.97 (t, $J = 7.4$ Hz, 3H) ppm. $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 174.9 (C), 138.2 (C), 129.2 (CH), 124.3 (CH), 119.9 (CH), 44.5 (CH), 27.6 (CH_2), 17.7 (CH_3), 12.1 (CH_3) ppm. HRMS (ESI) for $\text{C}_{11}\text{H}_{16}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 178.1226, found 178.1234.

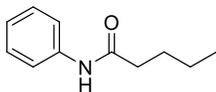
N-Phenylpivalamide (6d) [PMSA955]



The general procedure A was applied using *N,S*-diphenylthiocarbamate (**8**, 115 mg, 0.5 mmol) and *tert*-butyllithium (**5d**, 688 μL , 1.6 M solution in tetrahydrofuran, 1.1 mmol, 2.2 equiv). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 10% ethyl acetate in 25 min, 25 mL/min). *N*-Phenylpivalamide was obtained in 82% (73 mg) yield. Diphenyl disulfide was isolated in 95% (52 mg) yield. The spectroscopic data are in accordance with the literature.²¹

White solid, m.p.: 133-134 °C (lit.: 131-132 °C),²¹ $R_f = 0.15$ in heptane / EtOAc (9:1), $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.53 (d, $J = 7.7$ Hz, 2H), 7.32 (m, 3H), 7.11 (d, $J = 7.4$ Hz, 1H), 1.32 (s, 9H) ppm. $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 176.7 (C), 138.2 (C), 129.1 (CH), 124.3 (CH), 120.1 (CH), 39.8 (C), 27.8 (CH_3) ppm. HRMS (ESI) for $\text{C}_{11}\text{H}_{16}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 178.1226, found 178.1231.

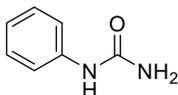
N-Phenylpentanamide (6e) [PMSA961]



The general procedure A was applied using *N,S*-diphenylthiocarbamate (**8**, 115 mg, 0.5 mmol) and *n*-butyllithium (**5e**, 440 μL , 2.5 M solution in tetrahydrofuran, 1.1 mmol, 2.2 equiv). The product was purified by an automated flash chromatography system using a heptane / ethyl acetate gradient (from 100% heptane to 40% ethyl acetate in 25 min, 25 mL/min). *N*-Phenylpentanamide was obtained in 65% (58 mg) yield. Diphenyl disulfide was isolated in 85% (46 mg) yield. The spectroscopic data are in accordance with the literature.²²

White solid, m.p.: 58-59 °C (lit.: 60-61 °C),²³ $R_f = 0.24$ in heptane / EtOAc (9:1), $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.51 (d, $J = 7.8$ Hz, 2H), 7.32 - 7.26 (m, 3H), 7.09 (t, $J = 7.2$ Hz, 1H), 2.35 (t, $J = 7.6$ Hz, 2H), 1.73 - 1.68 (m, 2H), 1.43 - 1.37 (m, 2H), 0.94 (t, $J = 7.3$ Hz, 3H) ppm. $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 171.6 (C), 138.2 (C), 129.1 (CH), 124.3 (CH), 120.0 (CH), 37.7 (CH_2), 27.8 (CH_2), 22.5 (CH_2), 13.9 (CH_3) ppm. HRMS (ESI) for $\text{C}_{11}\text{H}_{16}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 178.1226, found 178.1236.

N-Phenylurea (7) [PMSA954]



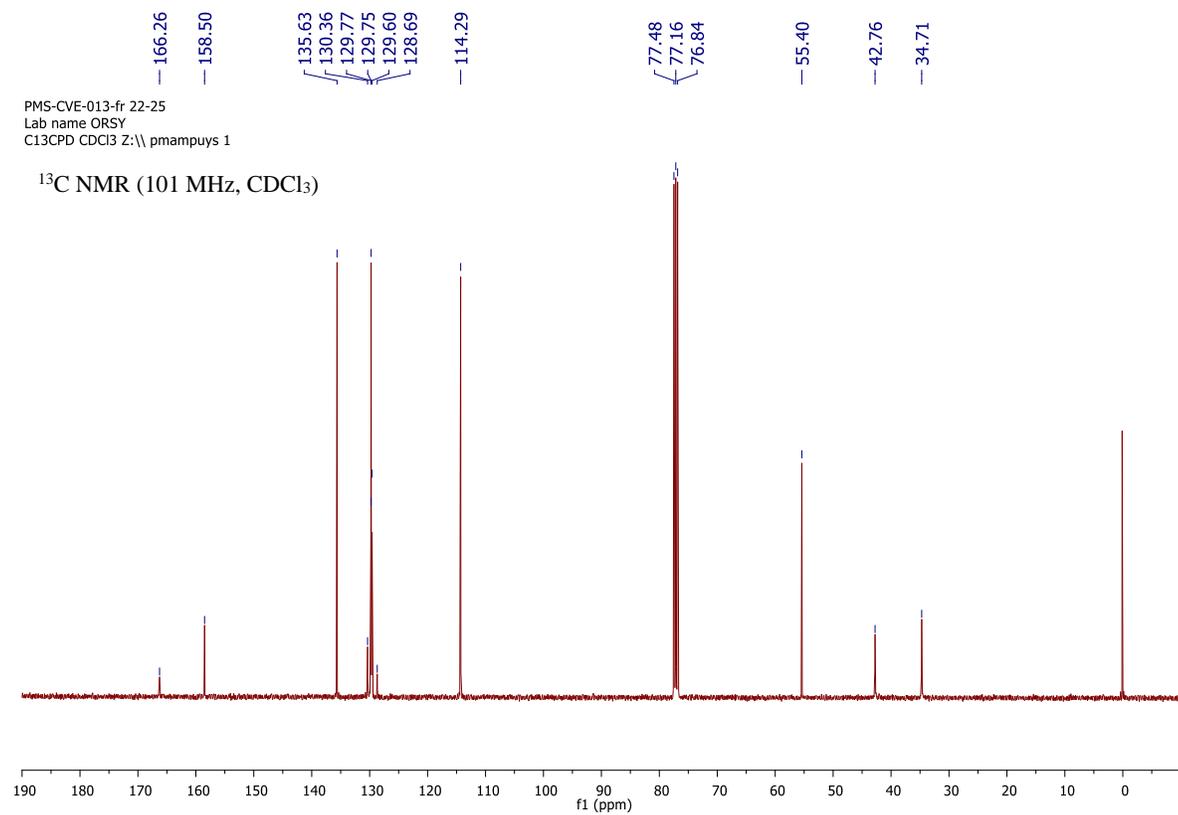
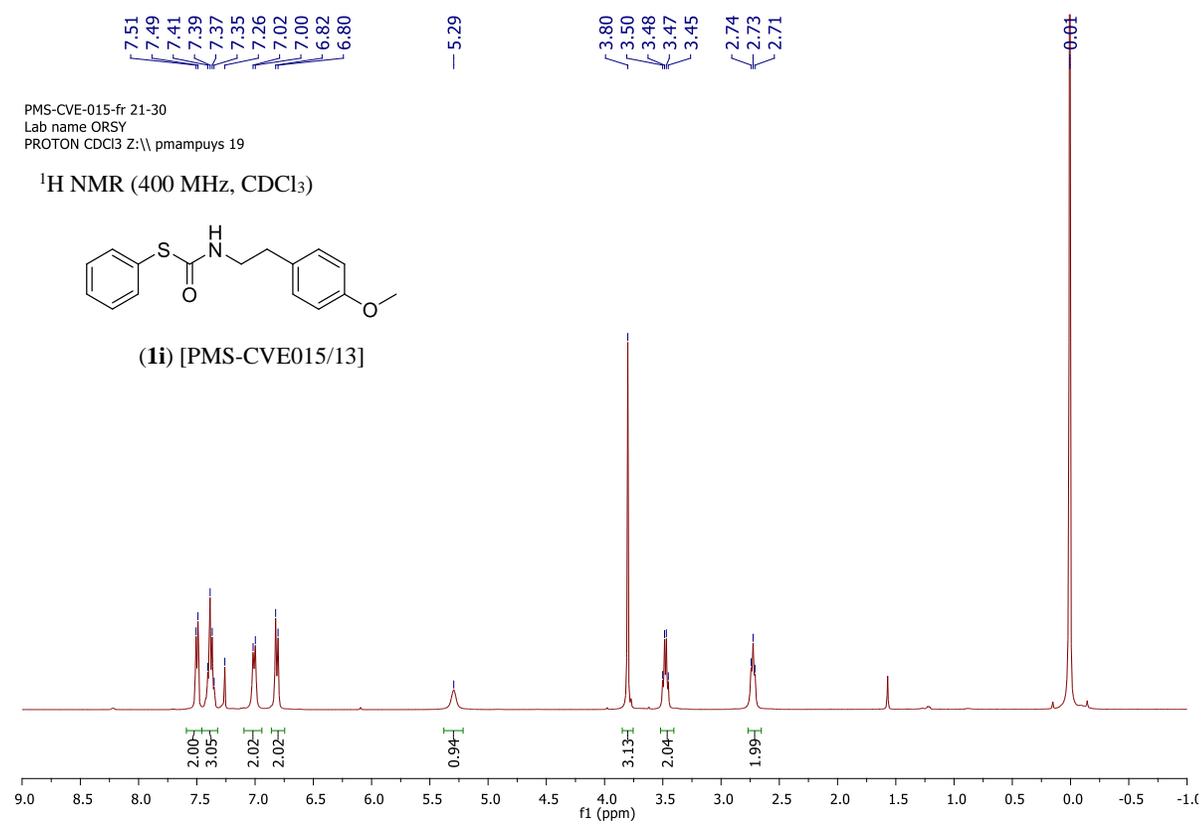
This compound was obtained as side product in the synthesis of **6a**, **c-e** with a Grignard reactant. The spectroscopic data are in accordance with the literature.²⁴

White solid, m.p.: 146-147 °C (lit.: 148 °C),²⁴ $R_f = 0.02$ in heptane / EtOAc (2:1), $^1\text{H NMR}$ (400 MHz, $\text{DMSO}-d_6$): δ 8.47 (br s, 1H), 7.38 (d, $J = 7.8$ Hz, 2H), 7.21 (t, $J = 7.9$ Hz, 2H), 6.88 (t, $J = 7.3$ Hz, 1H), 5.79 (br s, 2H) ppm. $^{13}\text{C NMR}$ (101 MHz, $\text{DMSO}-d_6$): δ 156.0 (C), 140.5 (C), 128.6 (CH), 121.0 (CH), 117.7 (CH) ppm. HRMS (ESI) for $\text{C}_7\text{H}_8\text{N}_2\text{ONa}$ $[\text{M}+\text{Na}]^+$ calcd. 159.0529, found 159.0539.

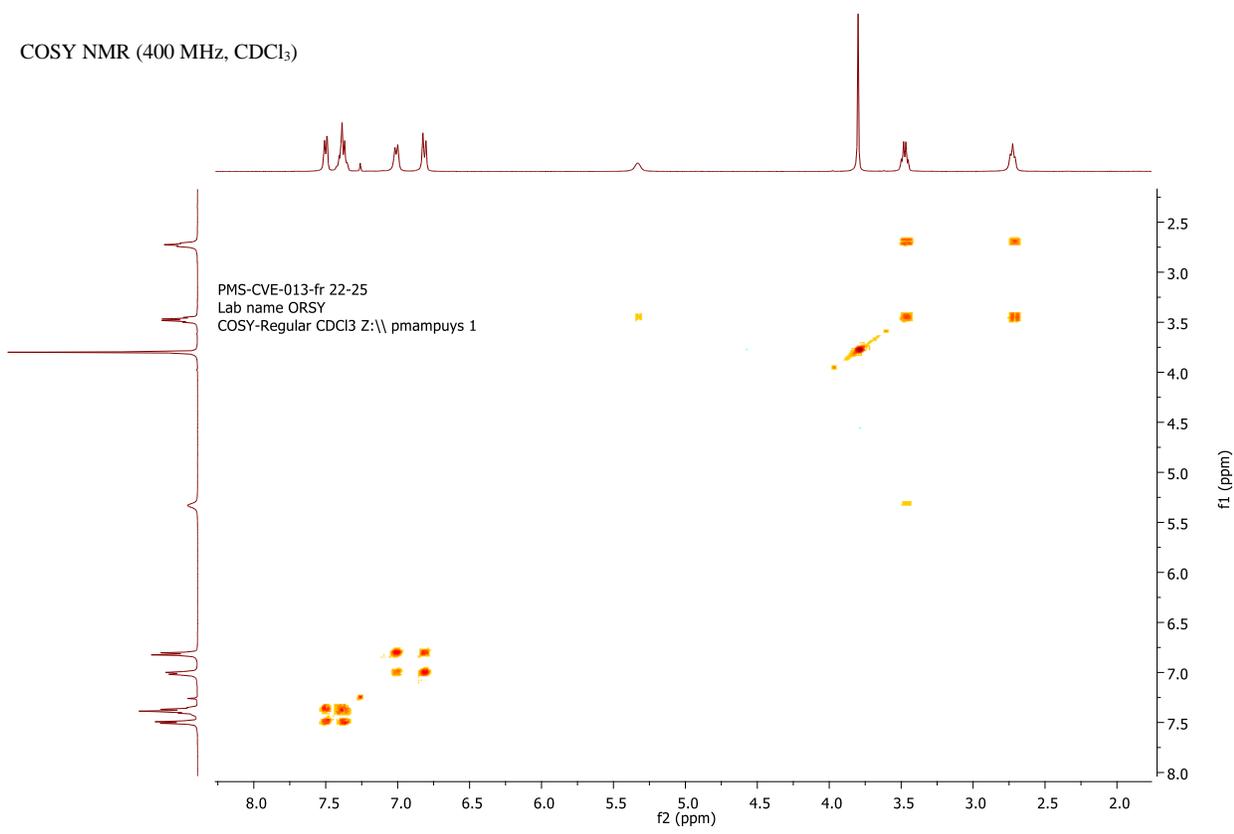
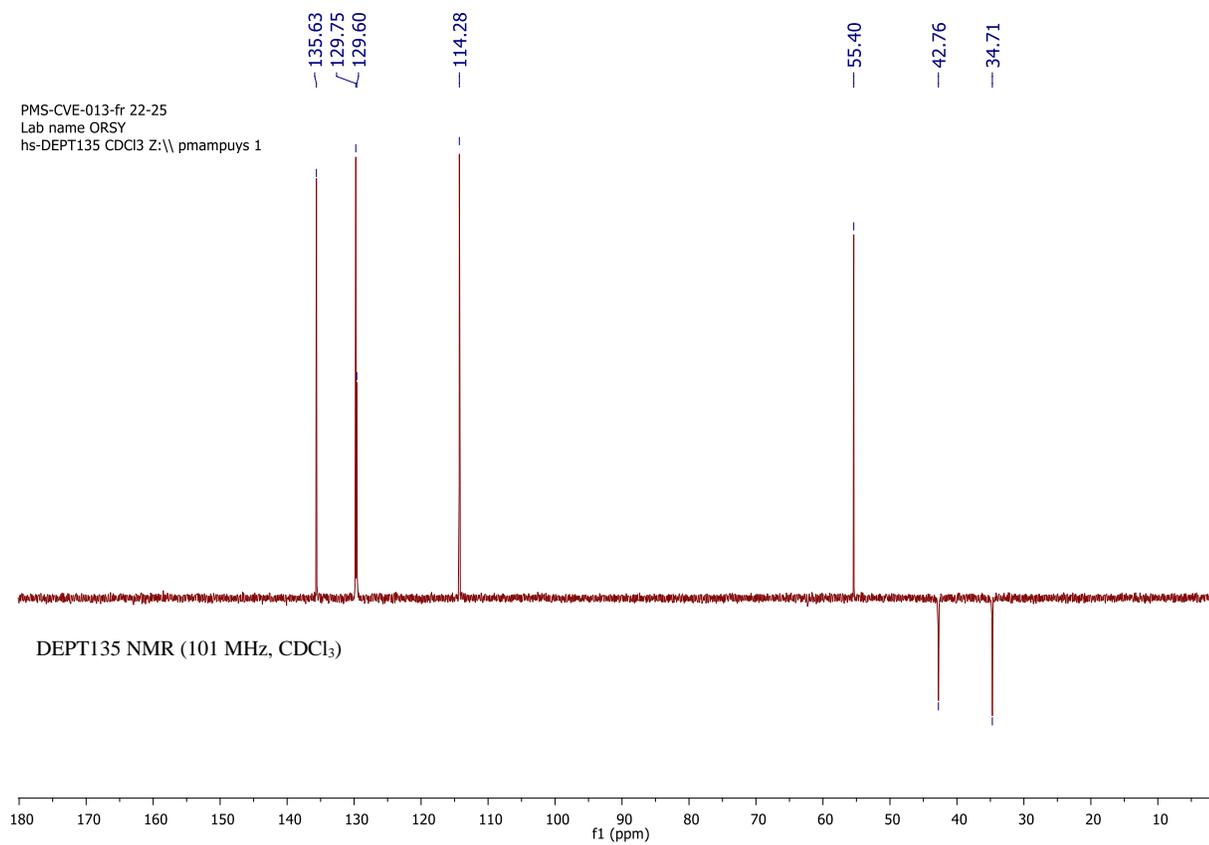
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8 Copies of the NMR spectra

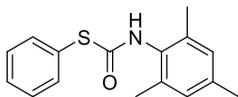


Copies of the NMR spectra

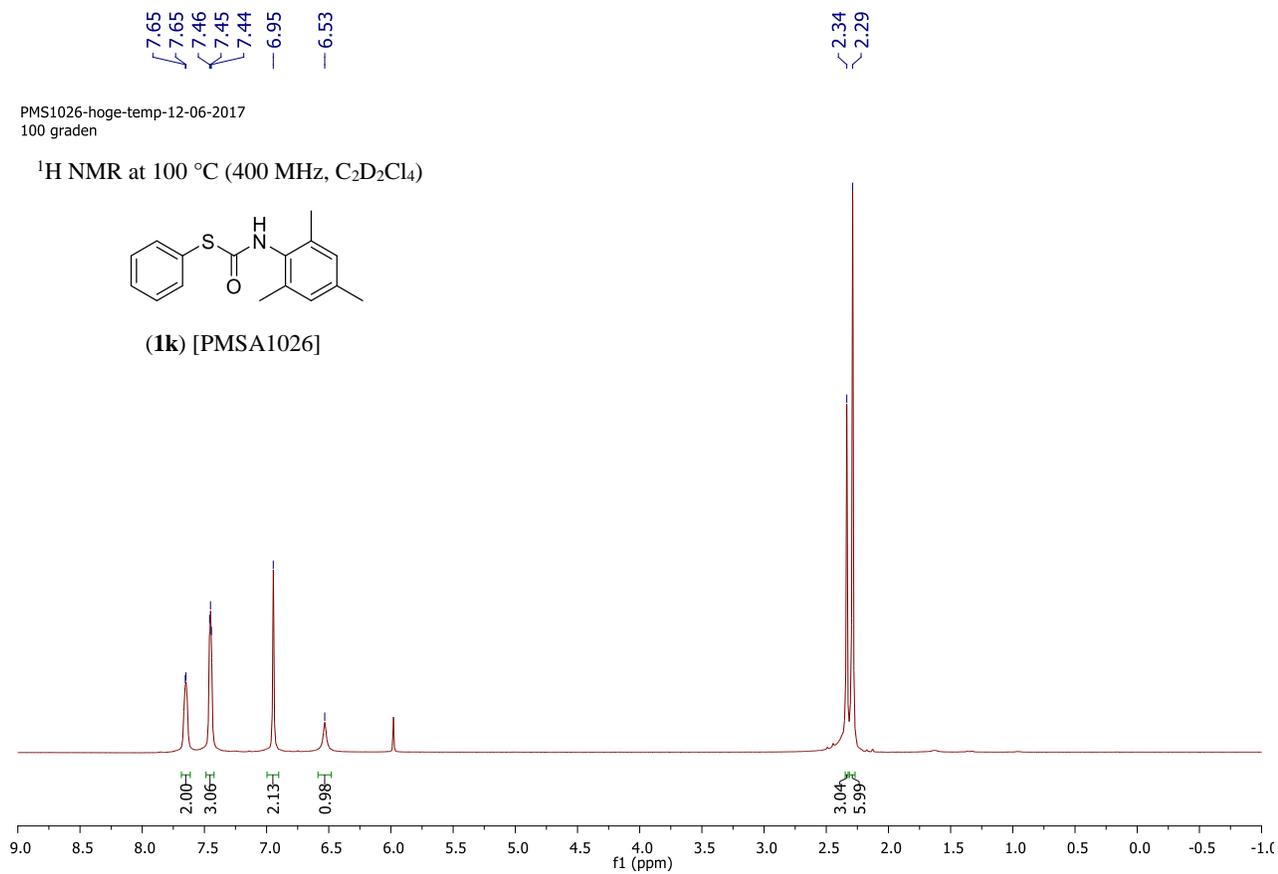


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^1H NMR at 100 °C (400 MHz, $\text{C}_2\text{D}_2\text{Cl}_4$)

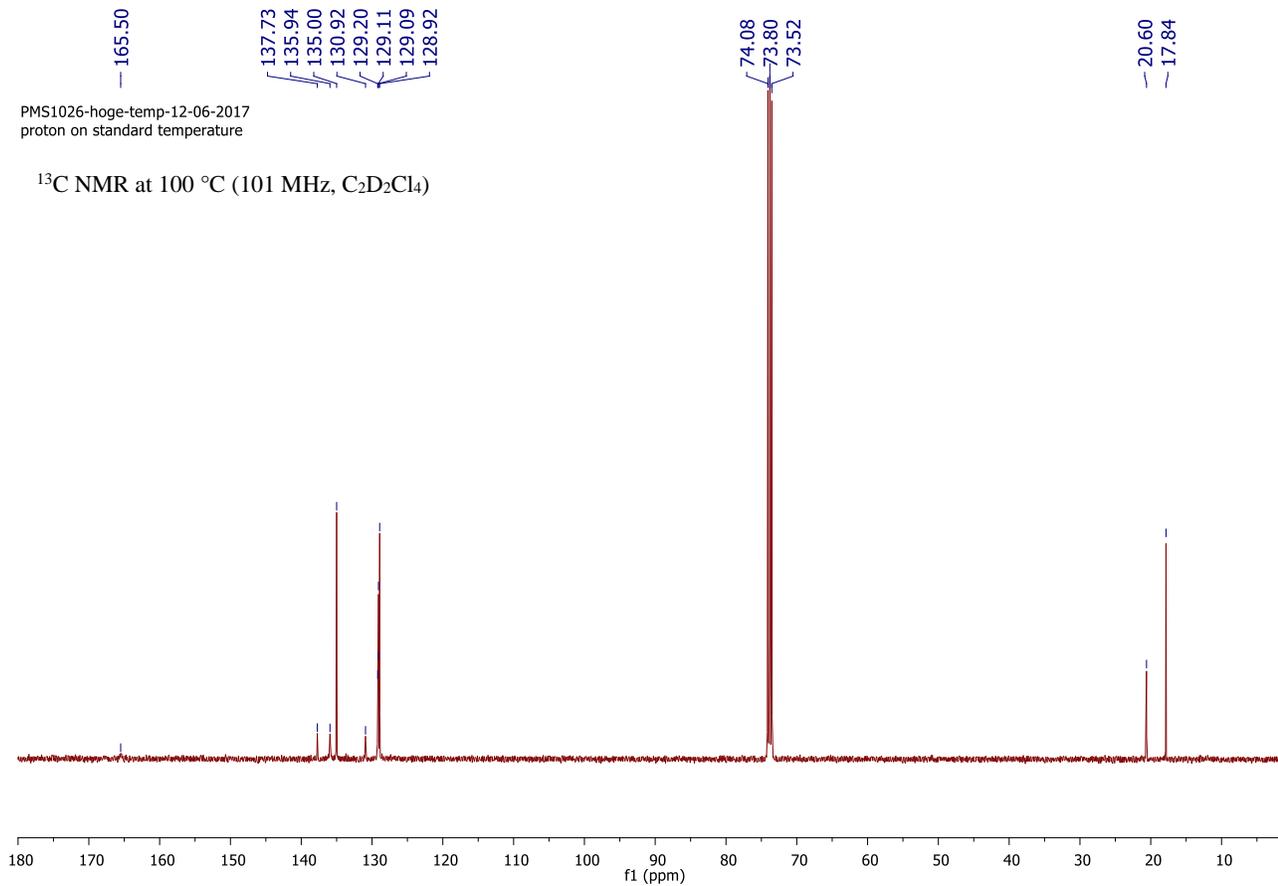


(1k) [PMSA1026]



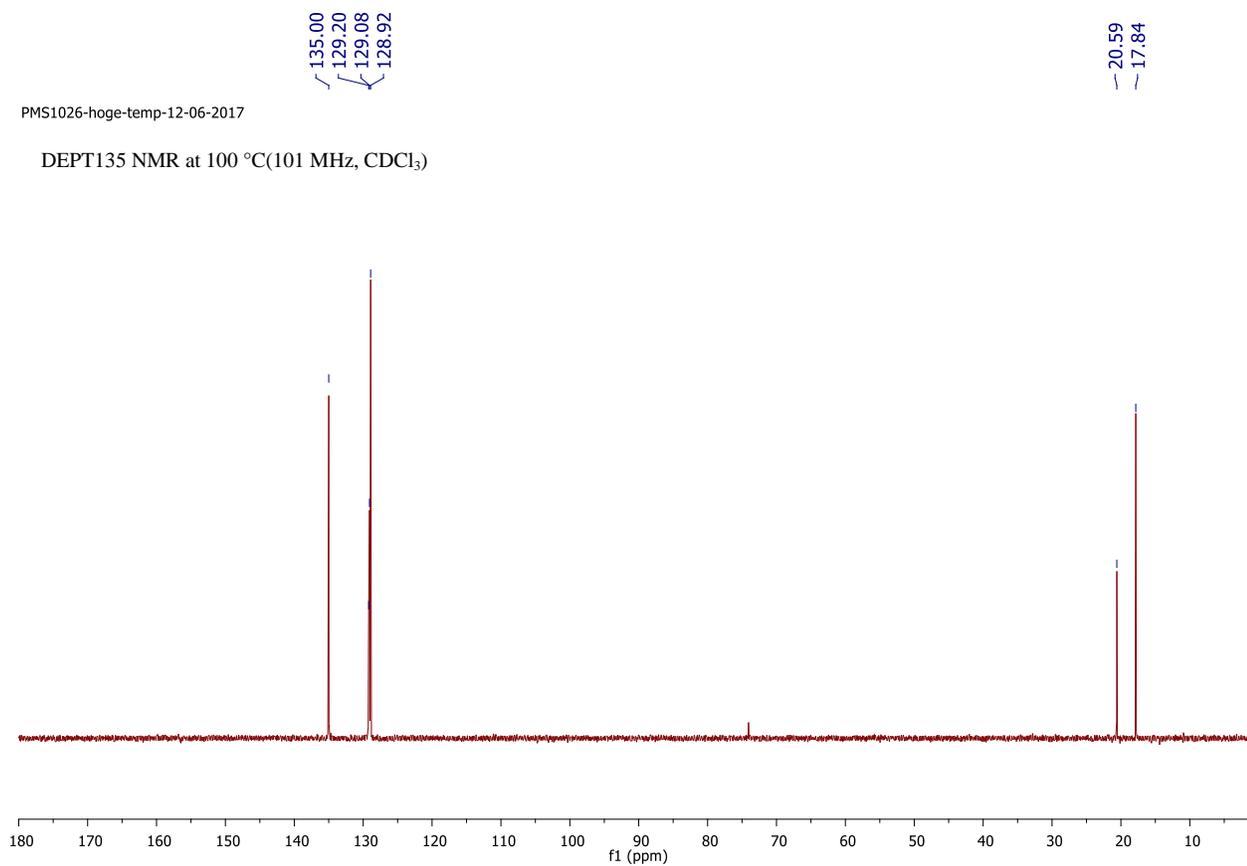
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proton on standard temperature

^{13}C NMR at 100 °C (101 MHz, $\text{C}_2\text{D}_2\text{Cl}_4$)

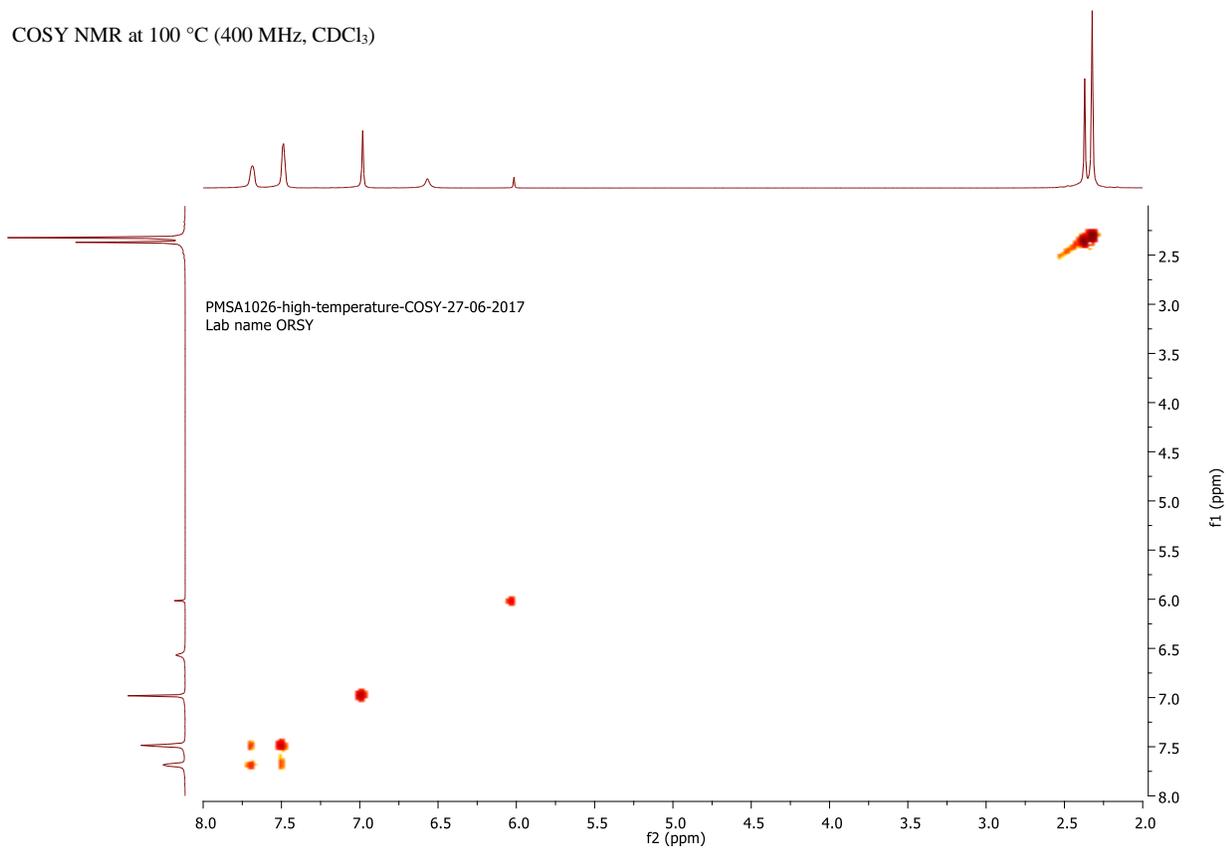


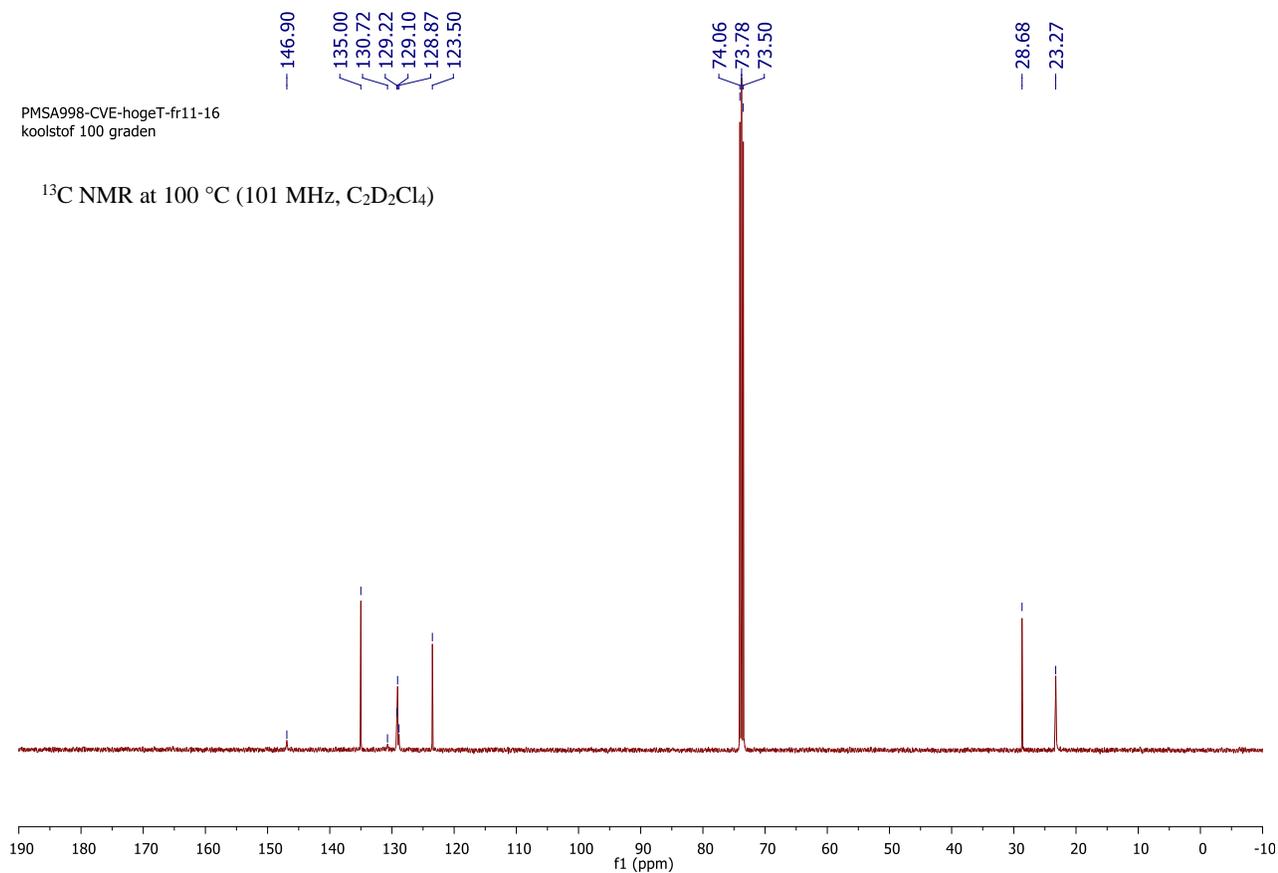
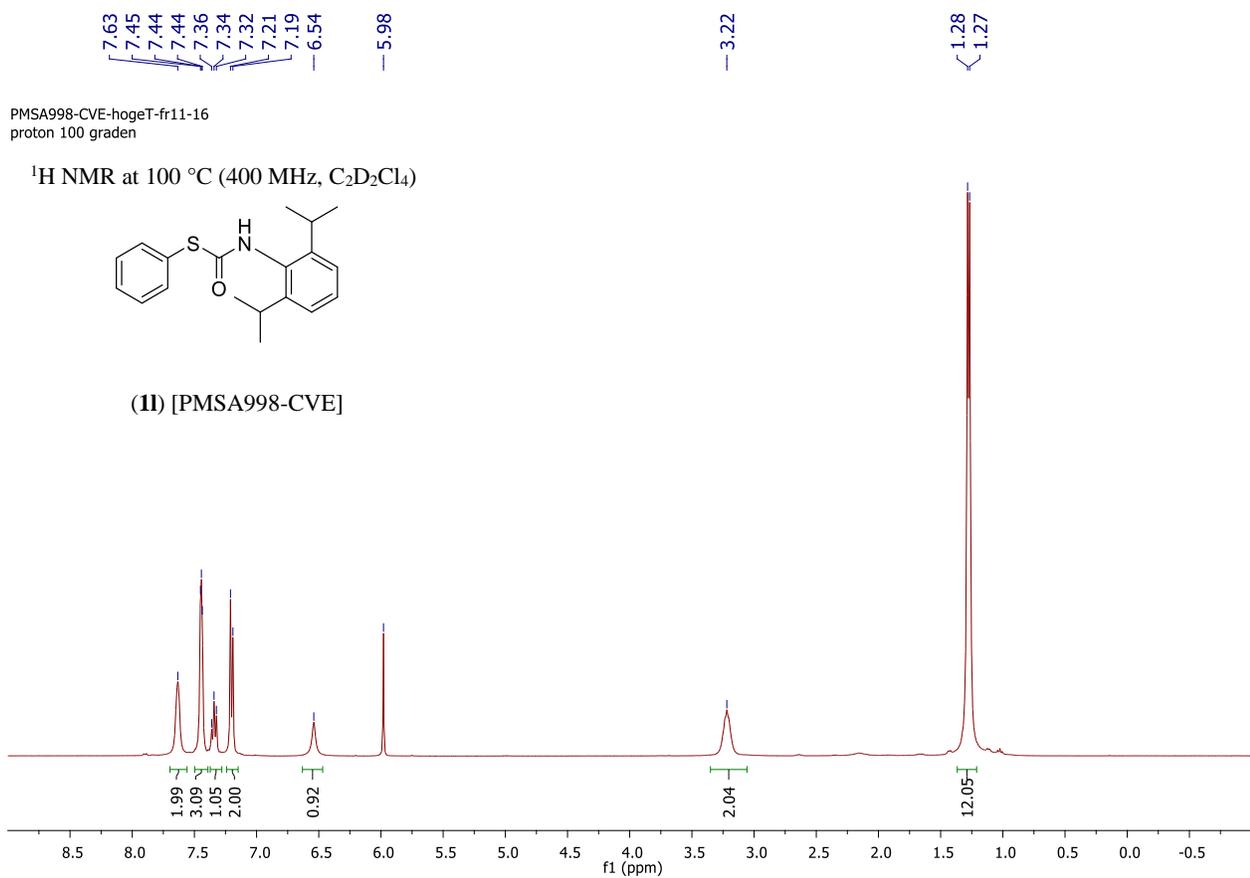
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DEPT135 NMR at 100 °C(101 MHz, CDCl₃)

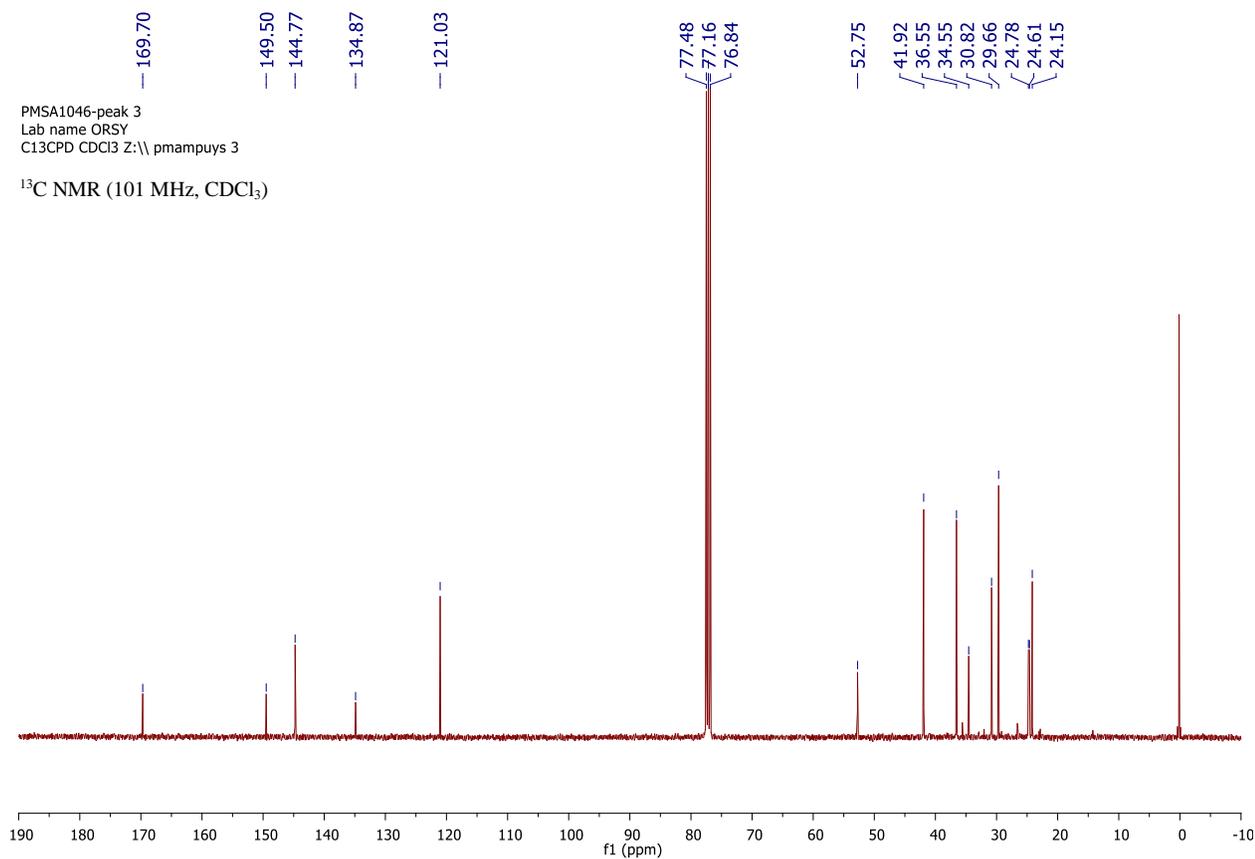
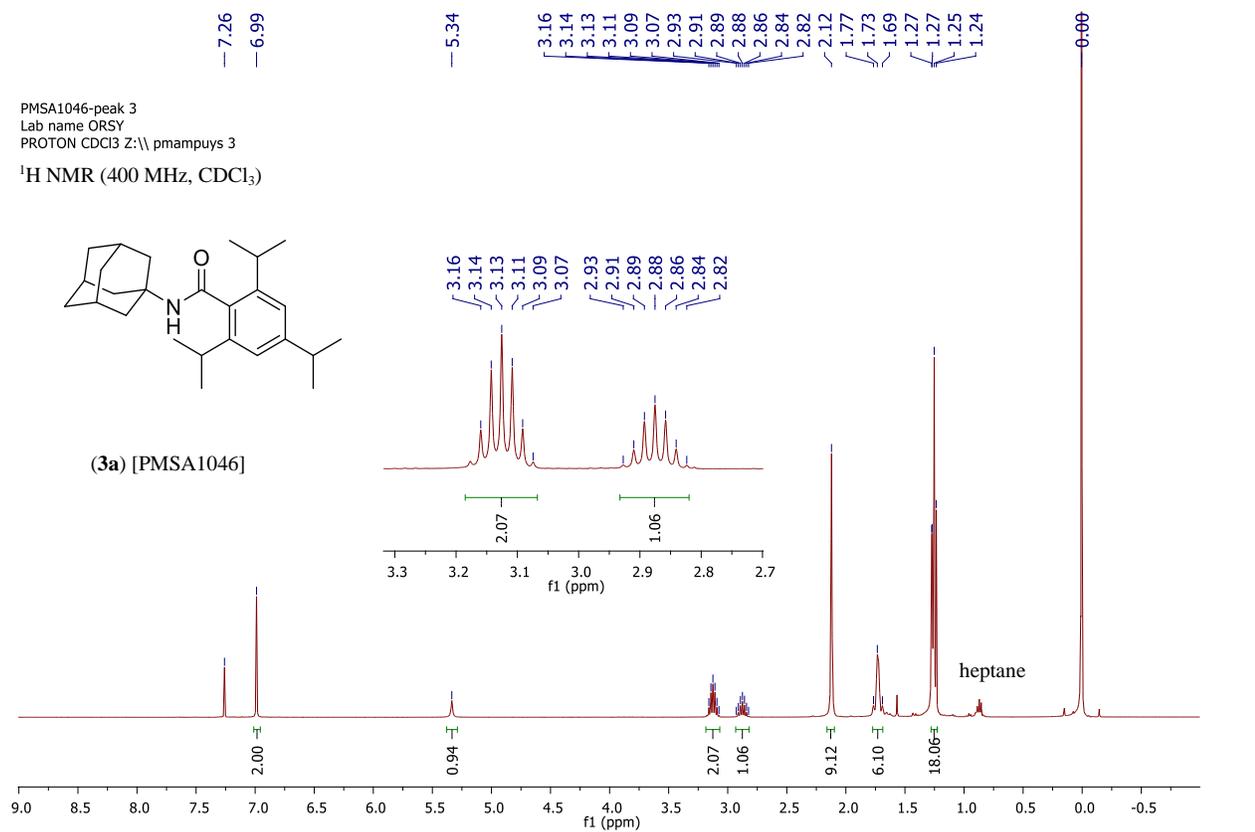


COSY NMR at 100 °C (400 MHz, CDCl₃)





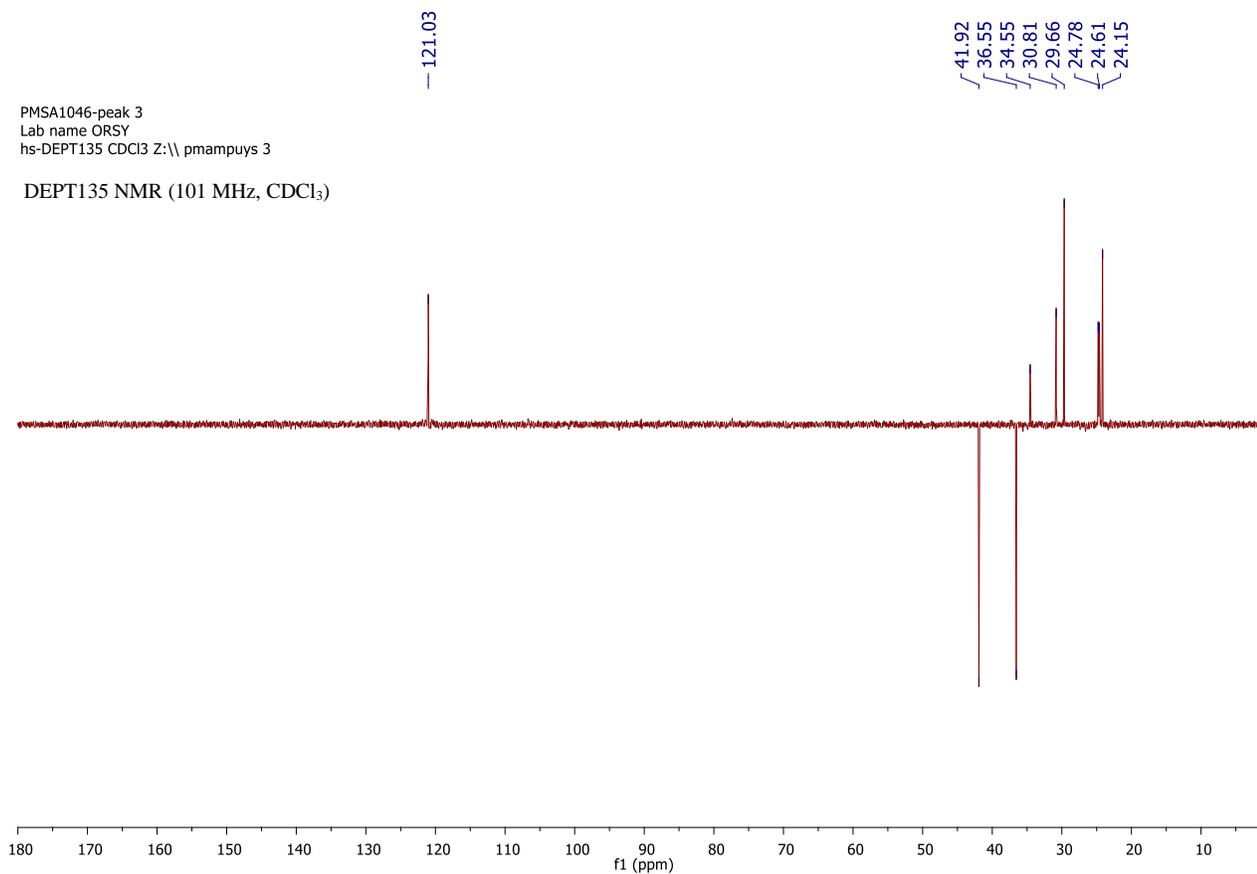
Copies of the NMR spectra



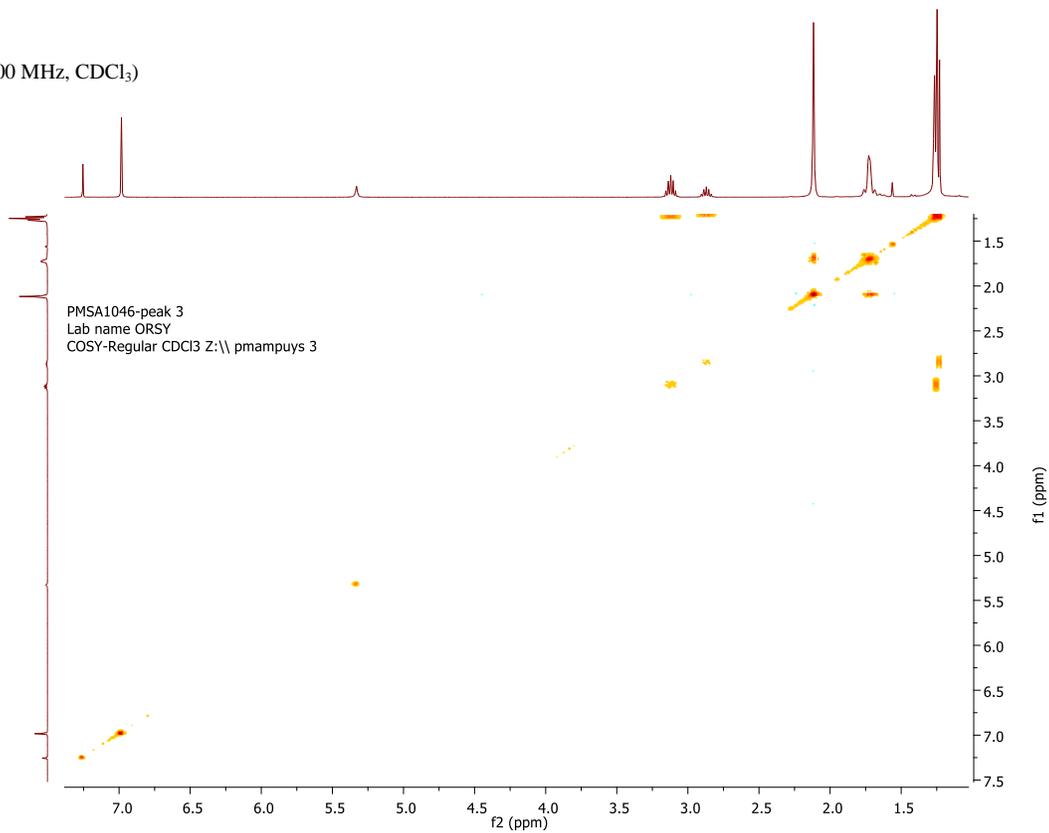
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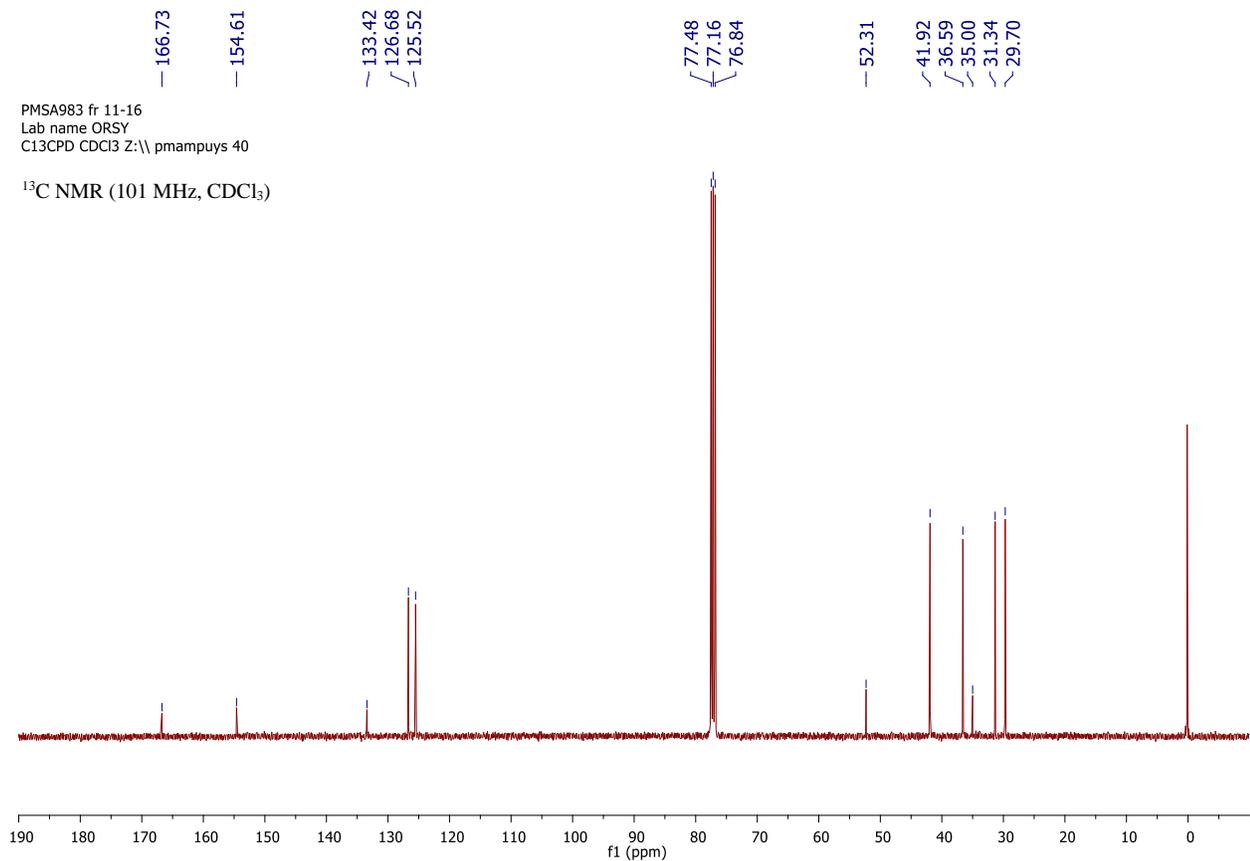
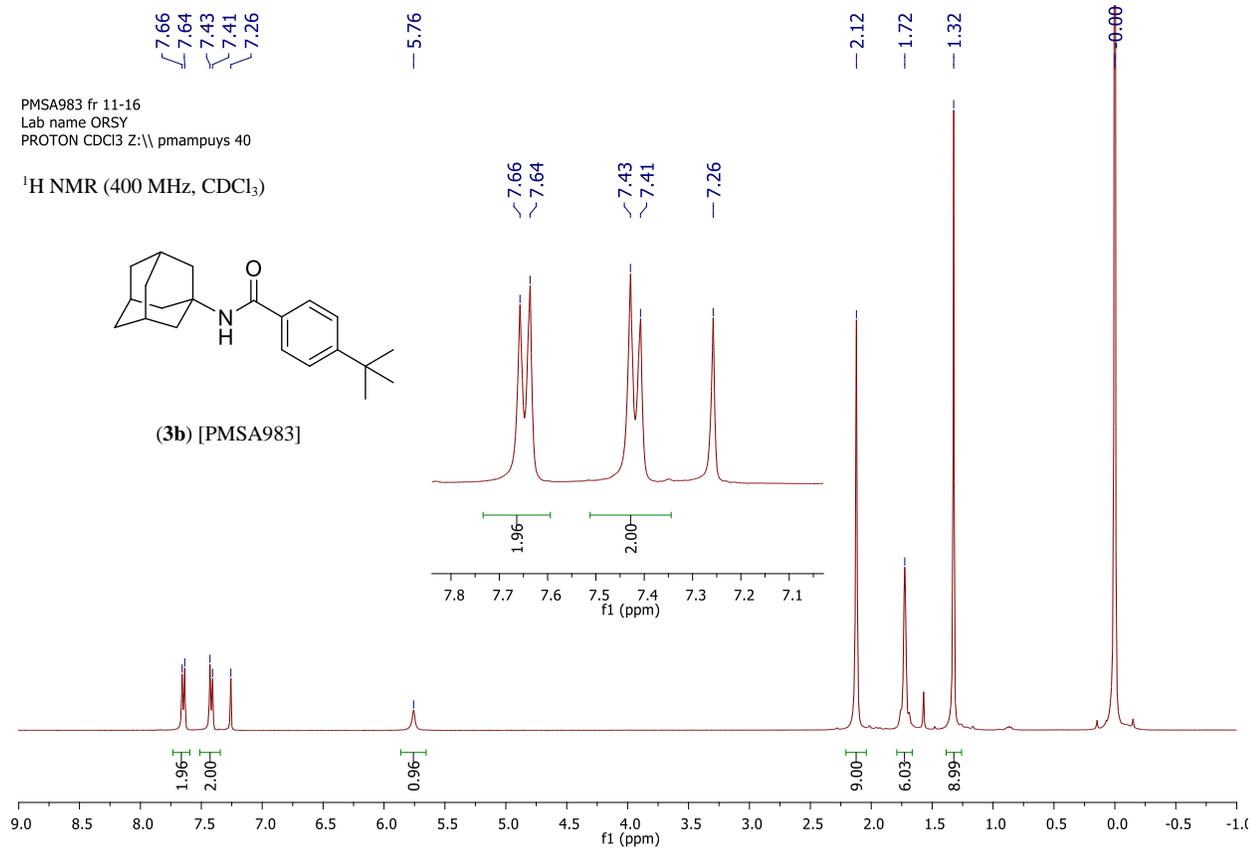
DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)



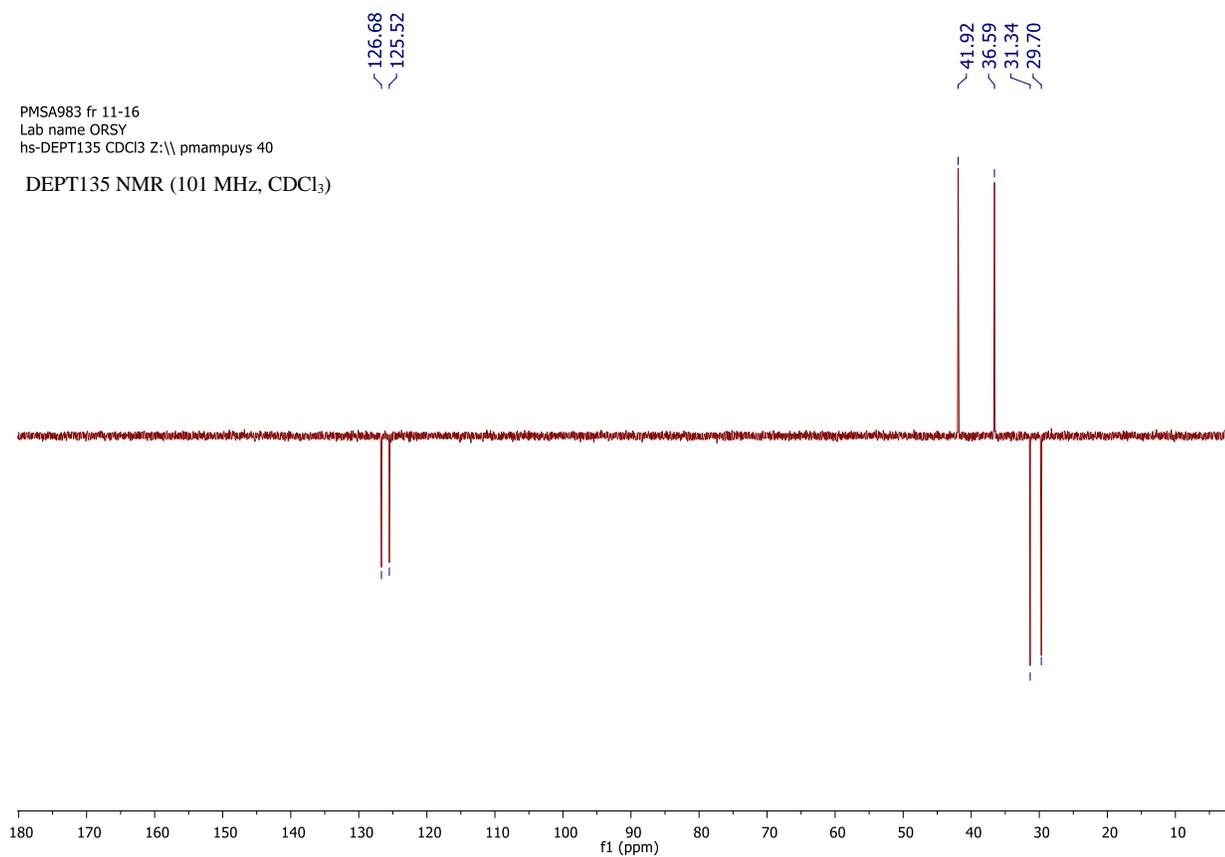
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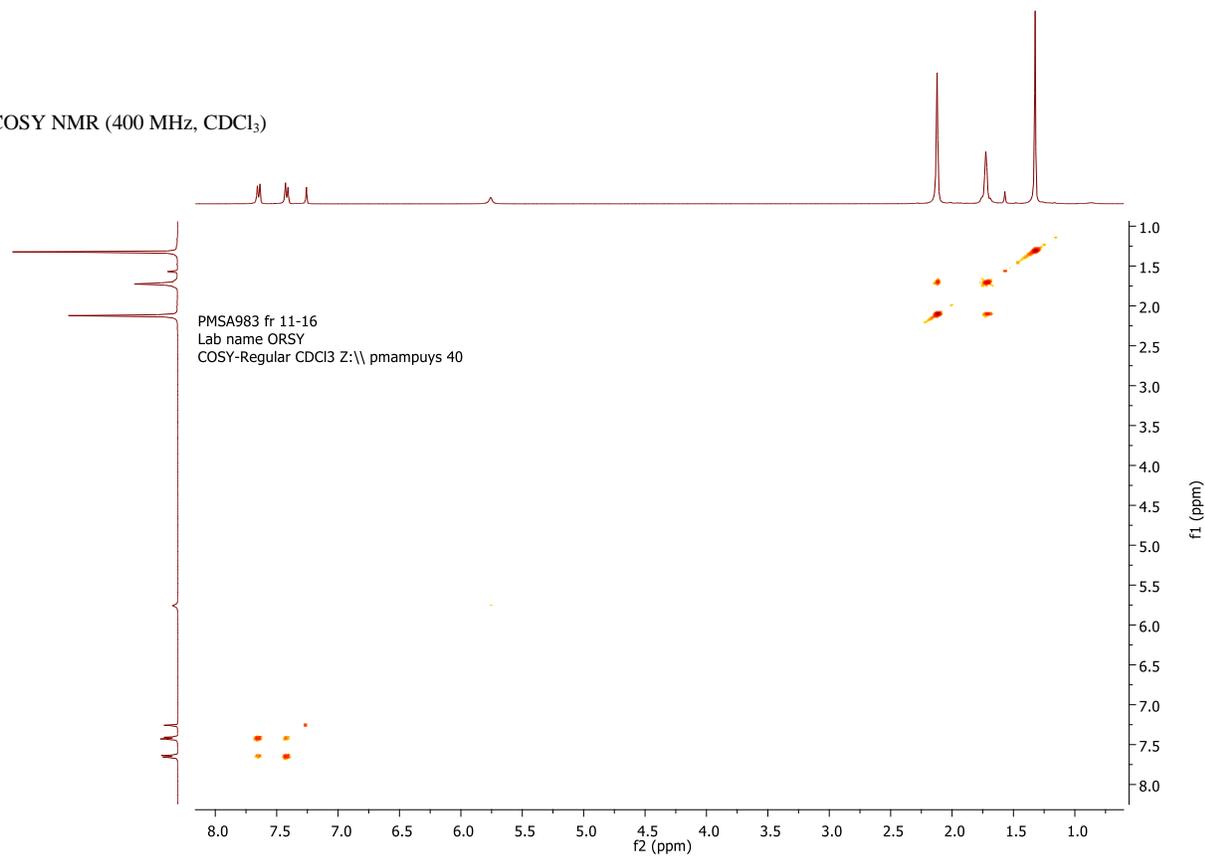
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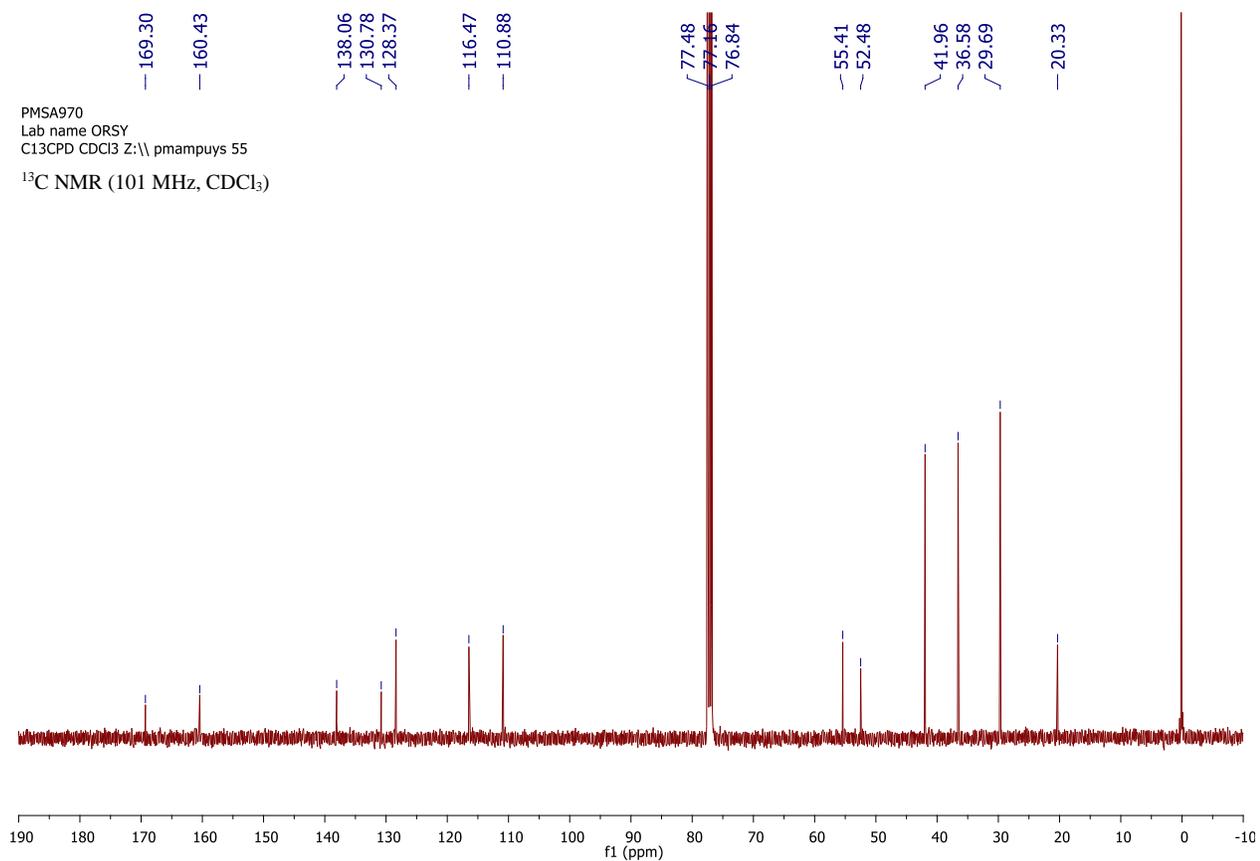
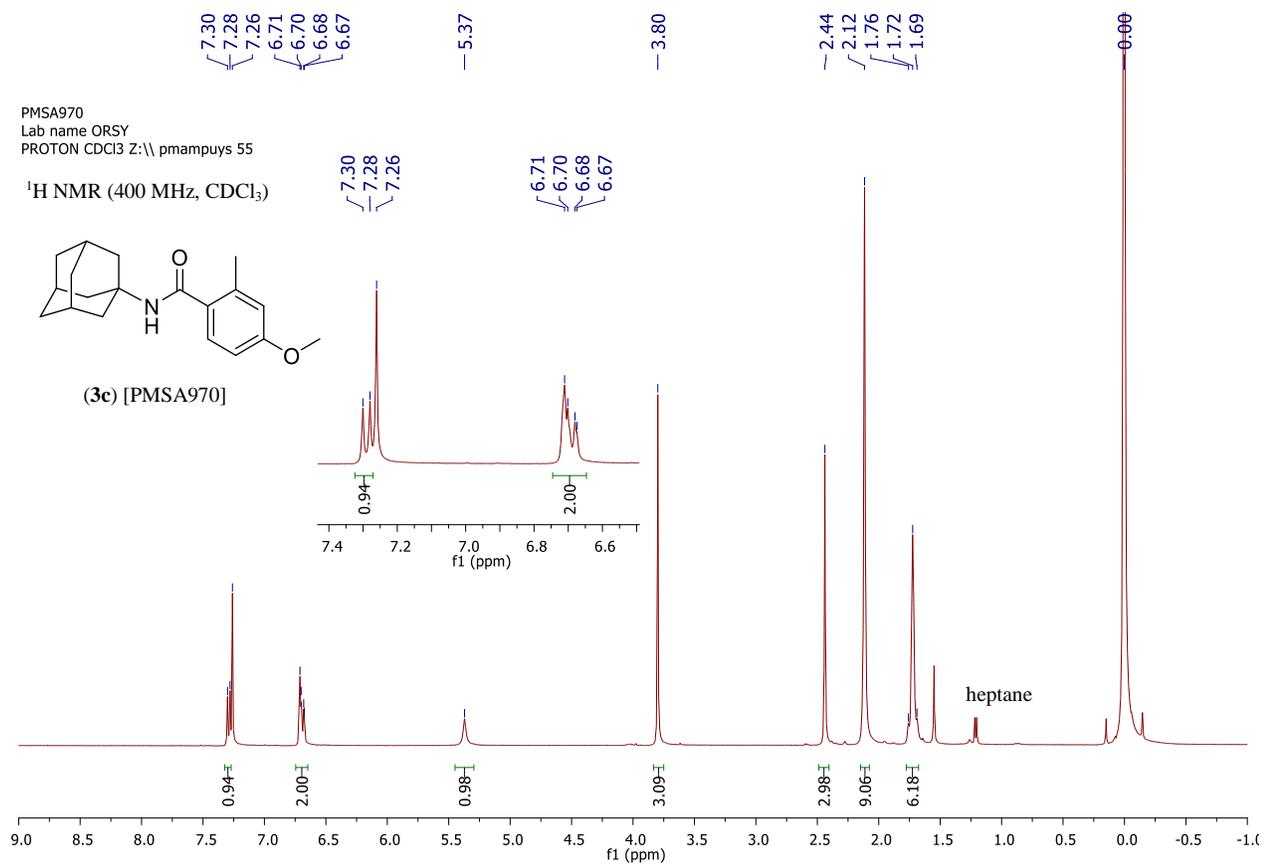
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COSY NMR (400 MHz, CDCl₃)



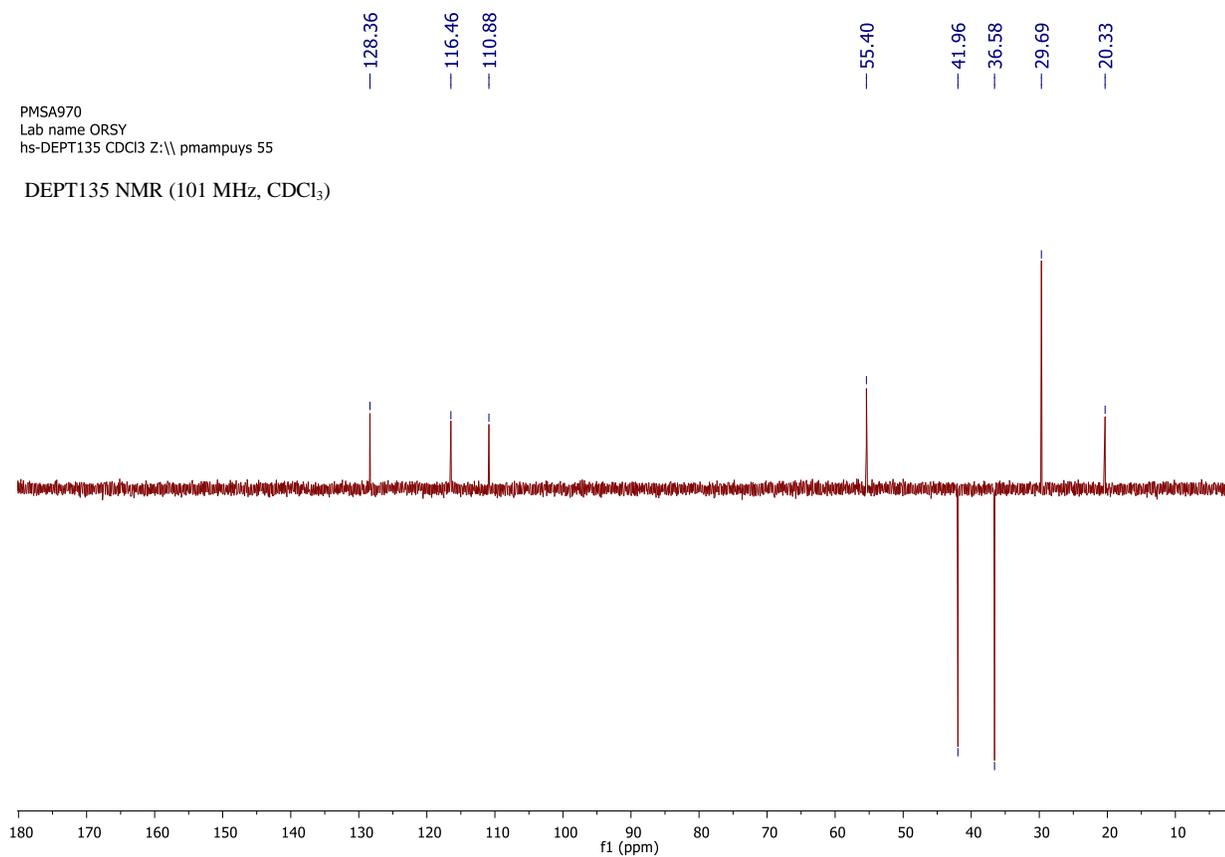
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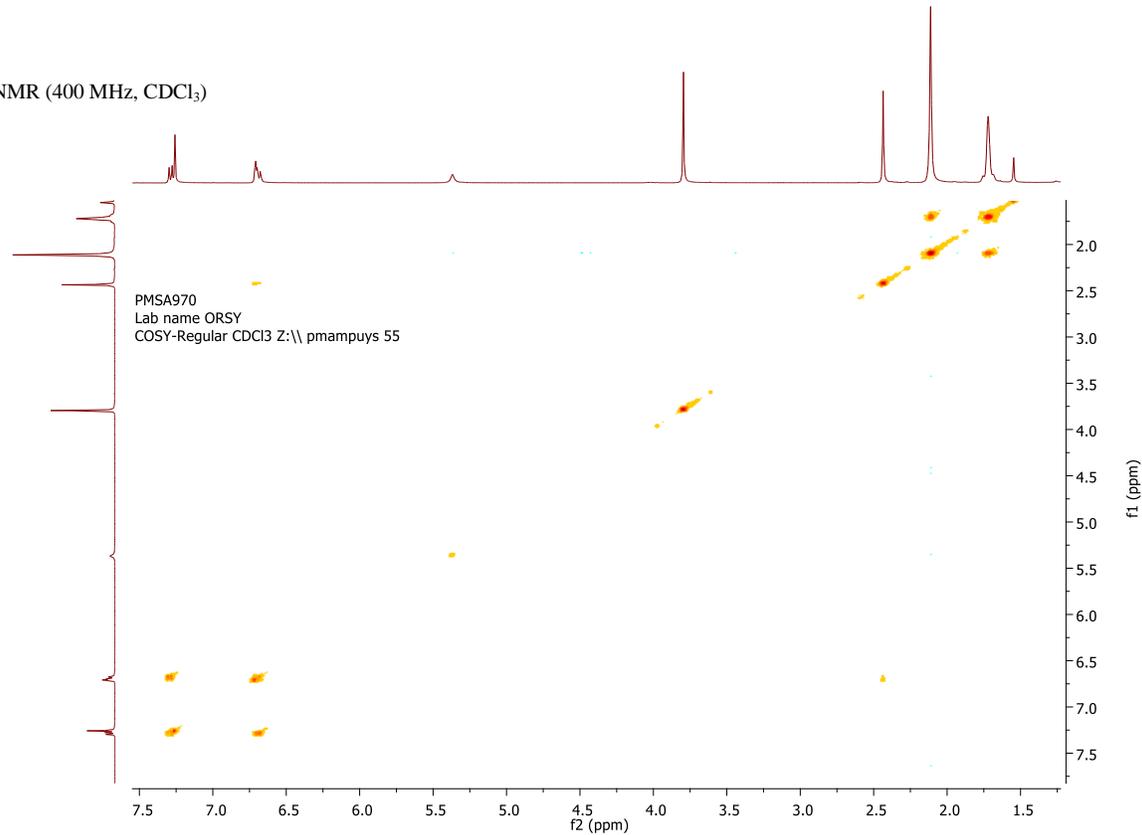
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PMSA970
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DEPT135 NMR (101 MHz, CDCl₃)

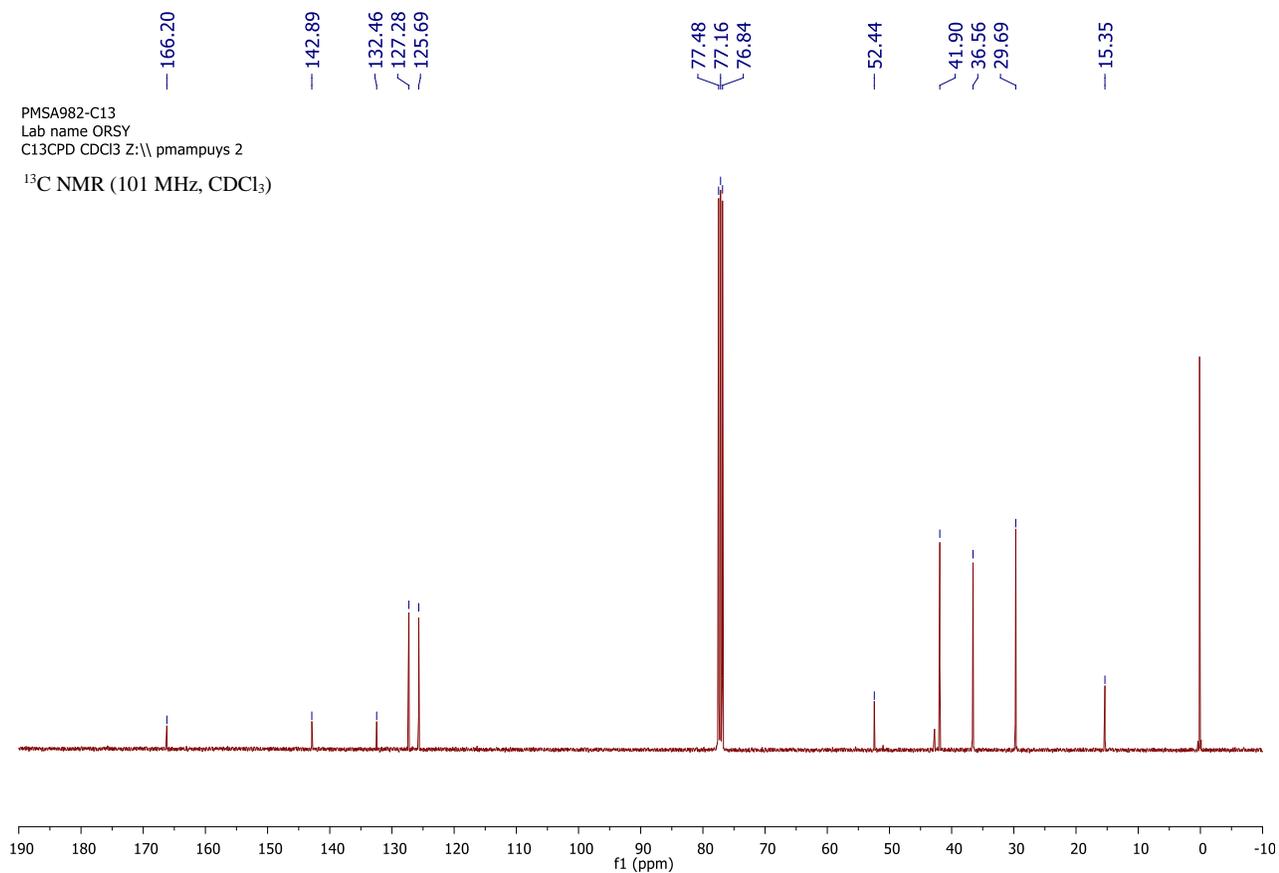
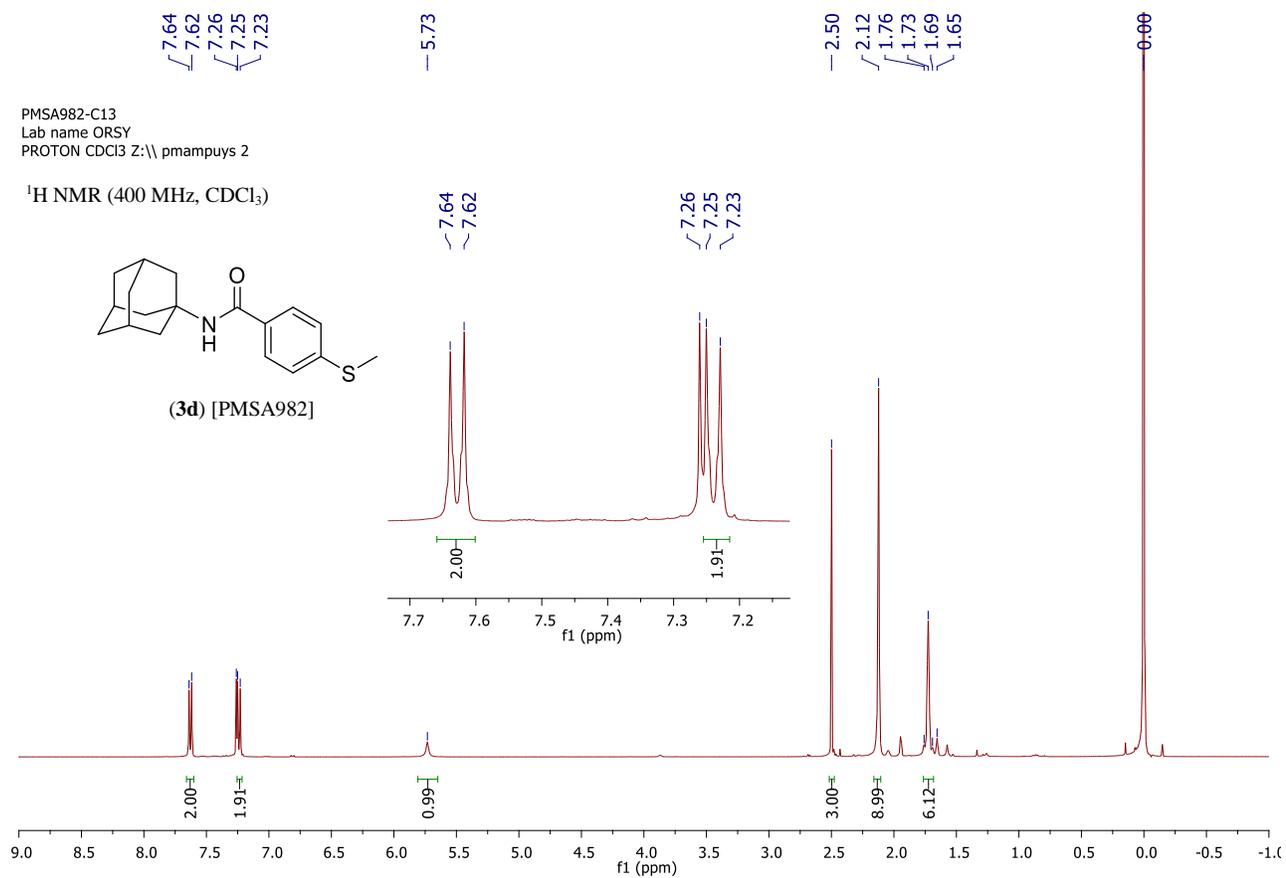


COSY NMR (400 MHz, CDCl₃)



PMSA970
Lab name ORSY
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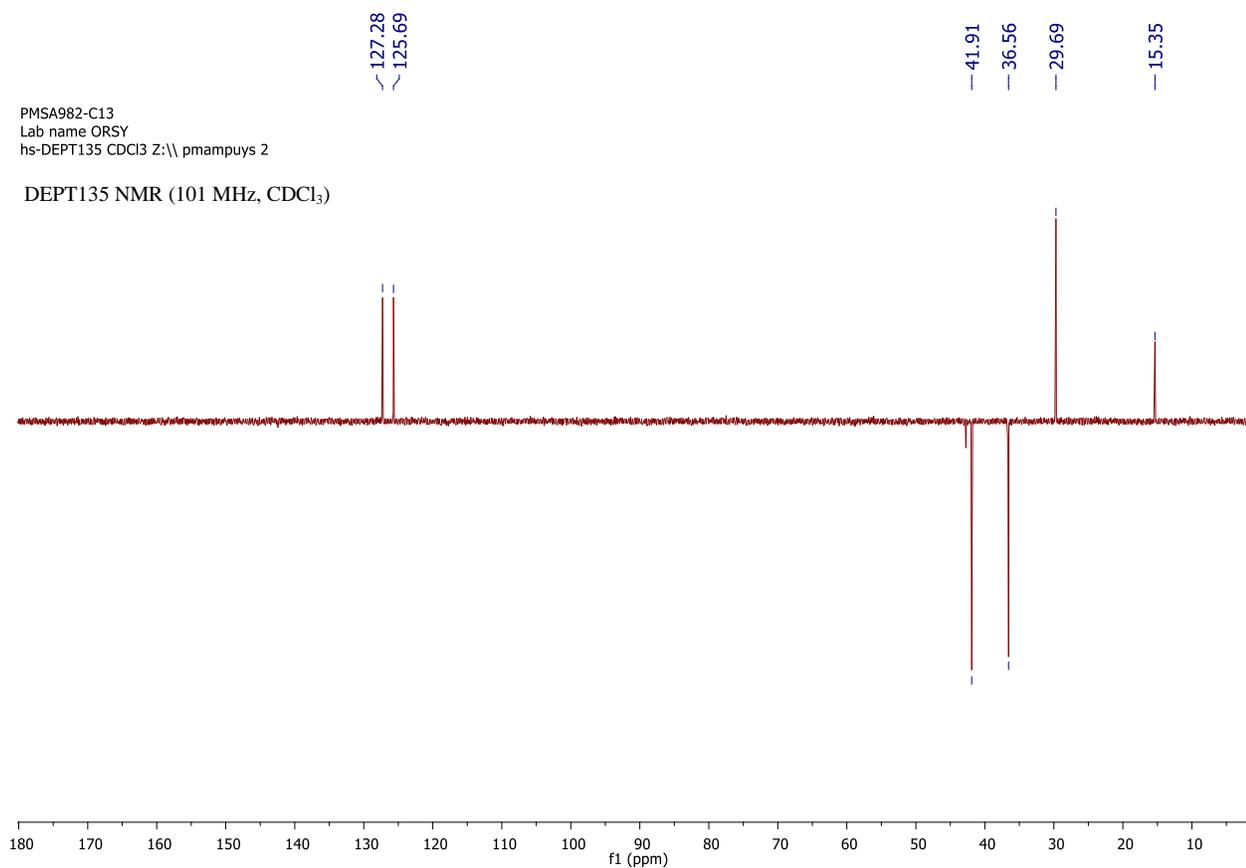
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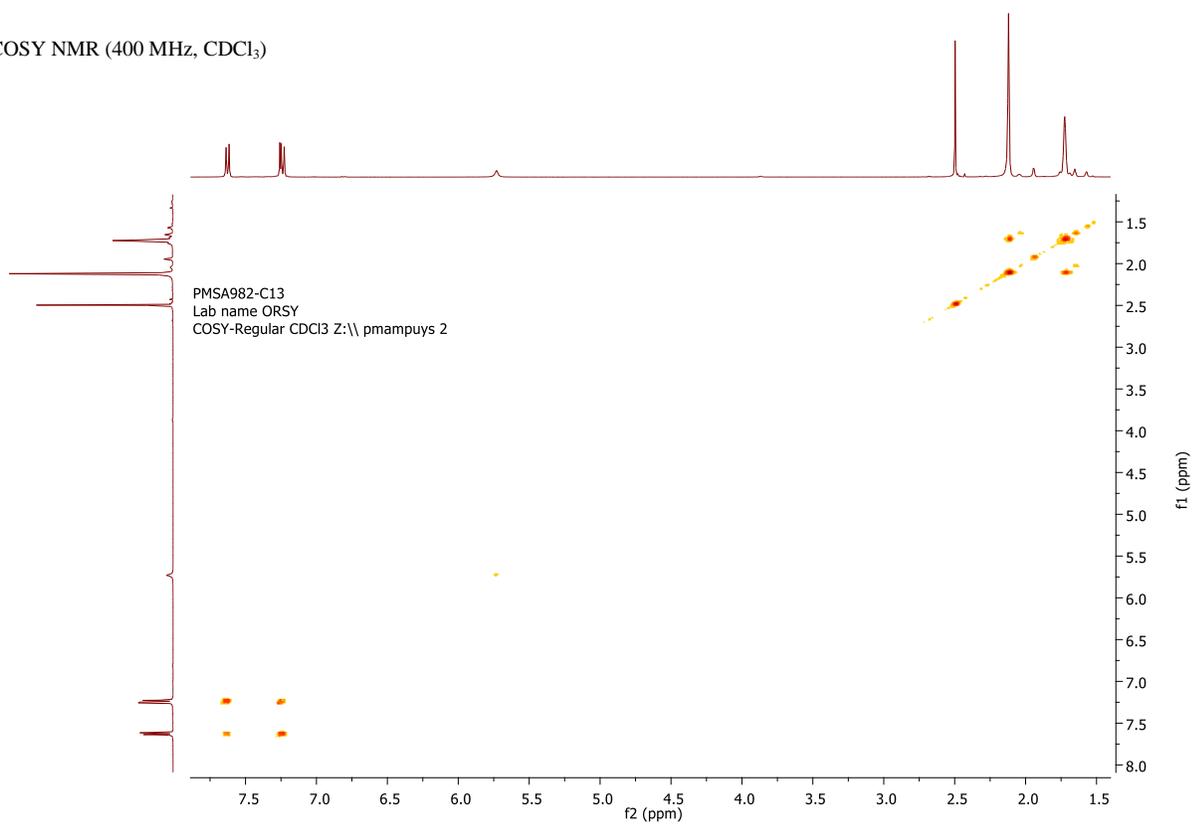
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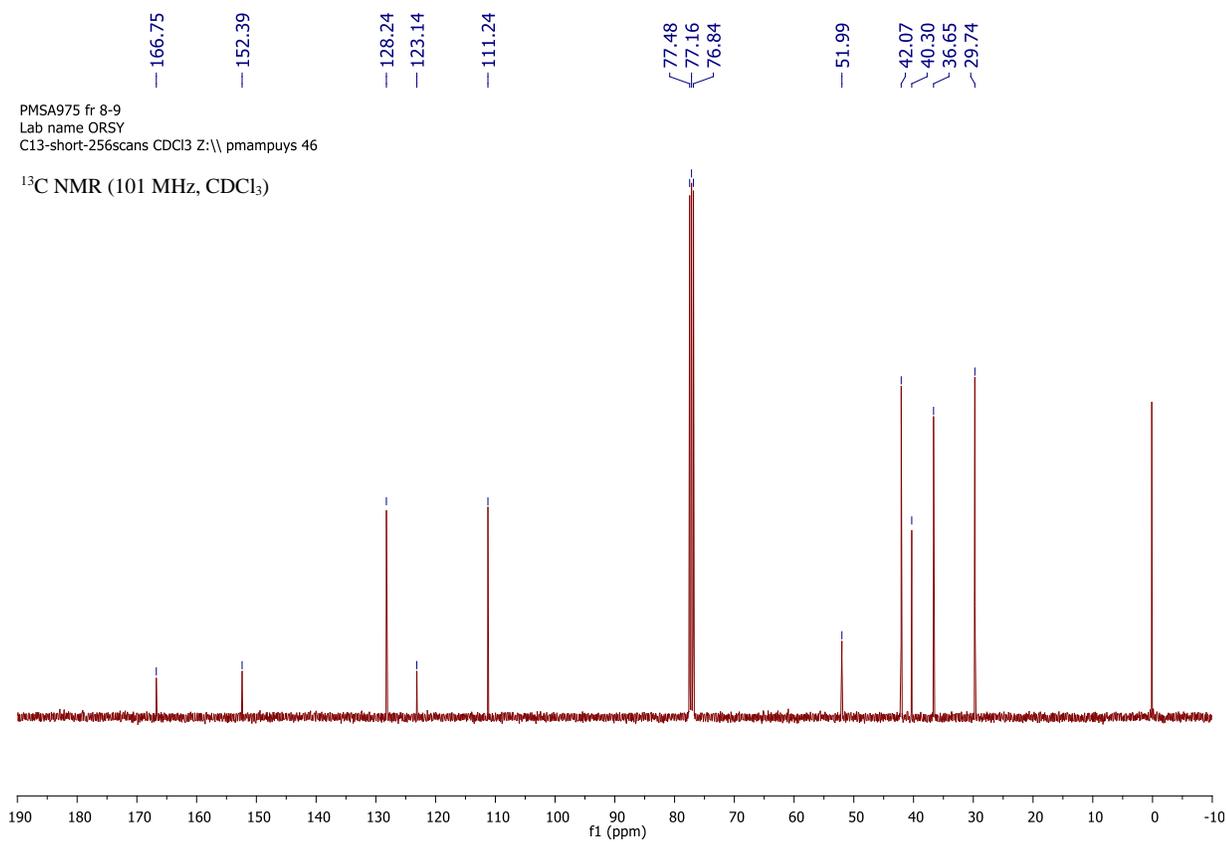
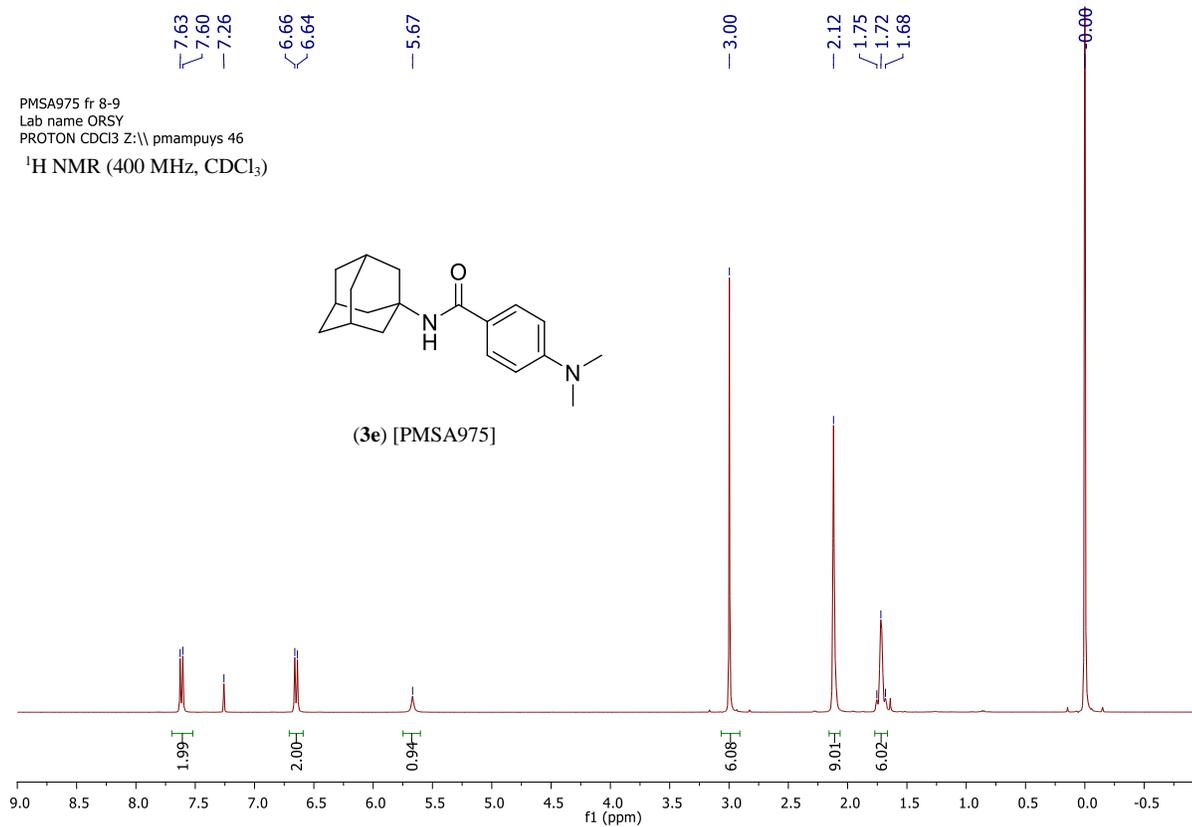
DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)



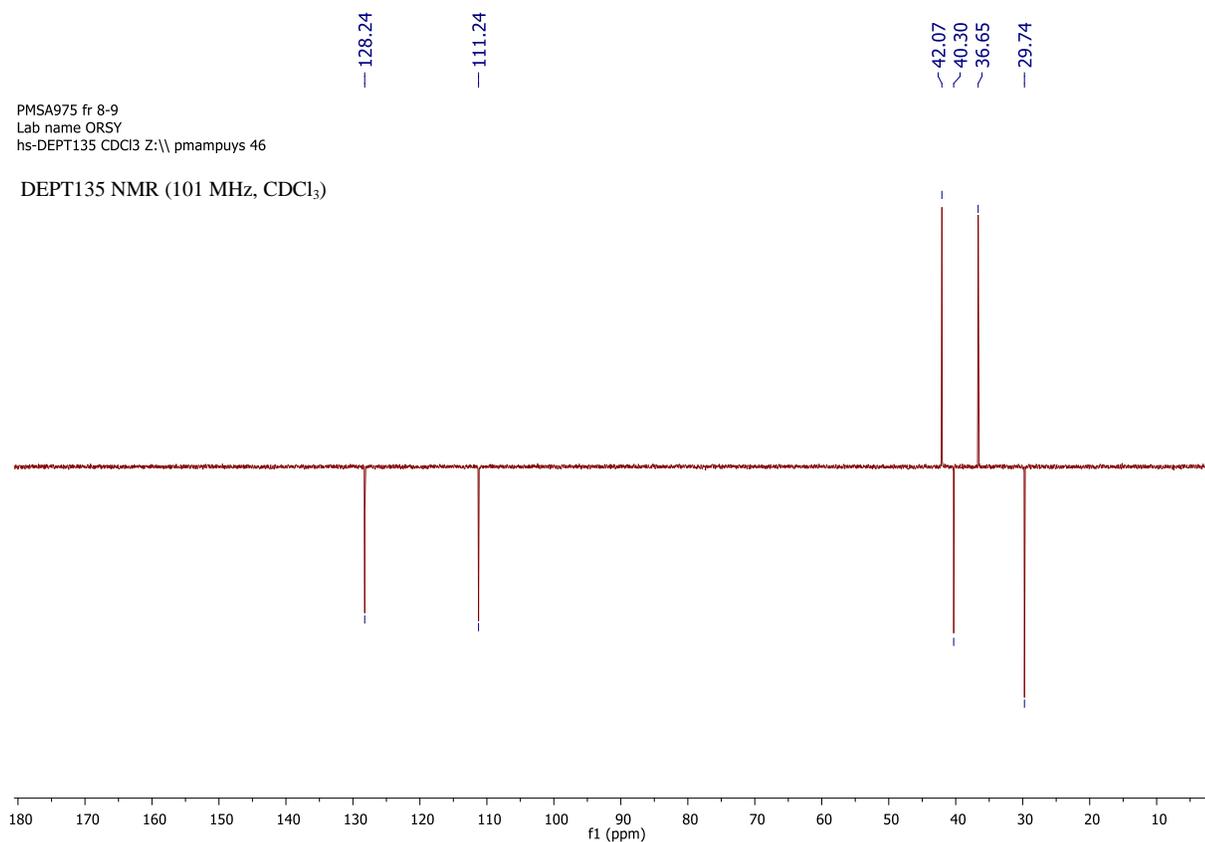
Copies of the NMR spectra



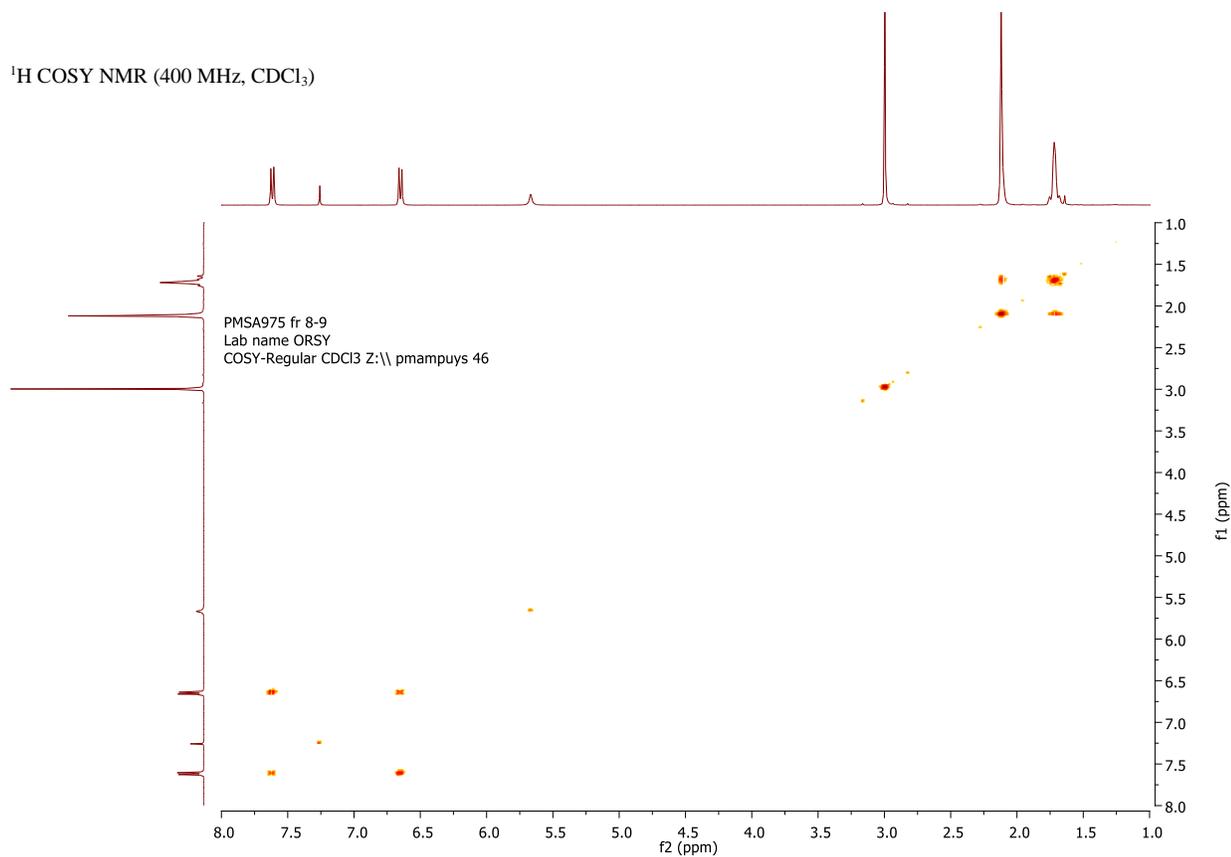
Copies of the NMR spectra

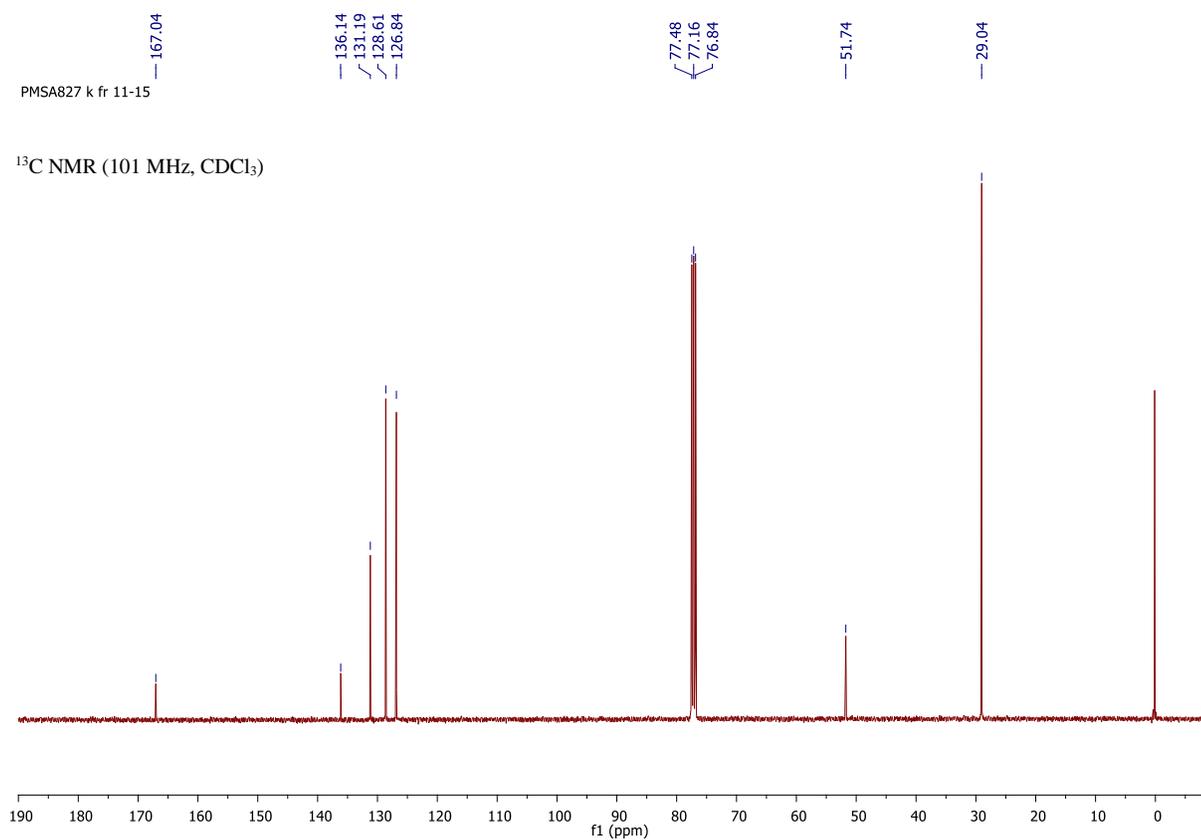
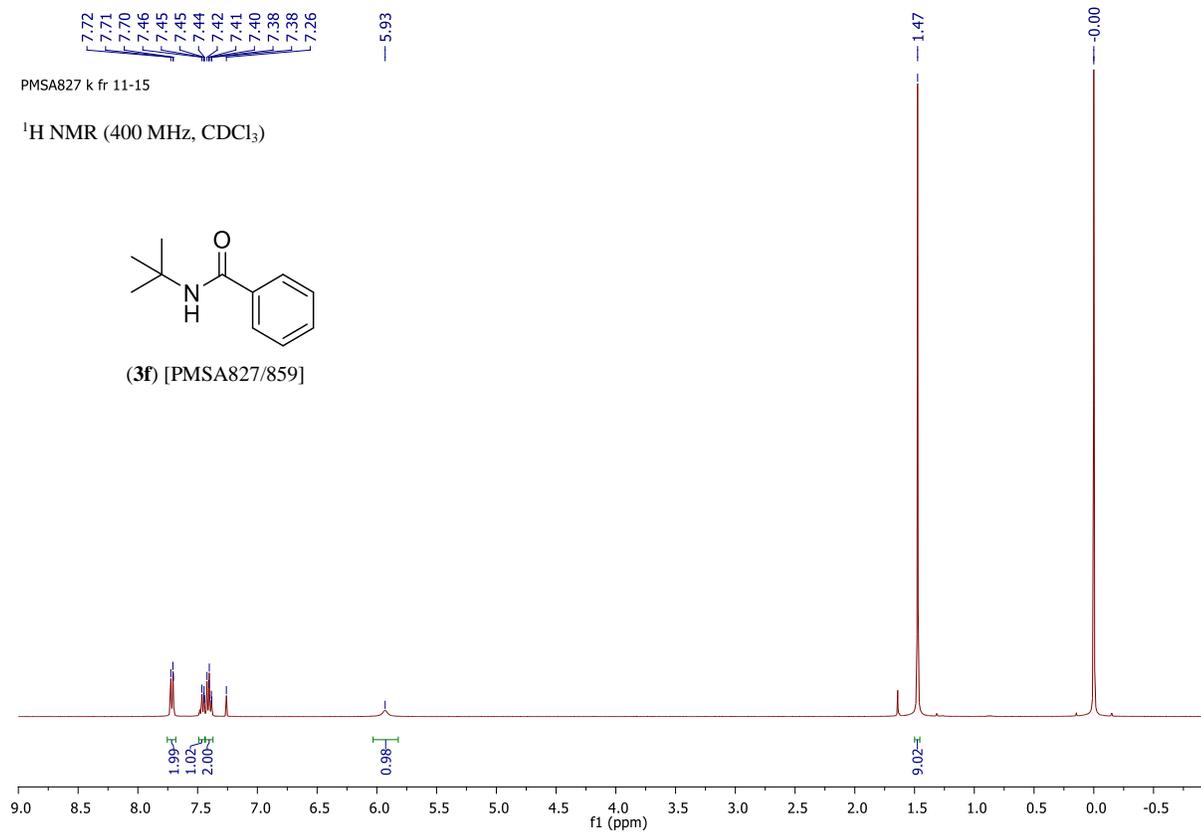
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Lab name ORSY
hs-DEPT135 CDCl₃ Z:\\ pmampuys 46

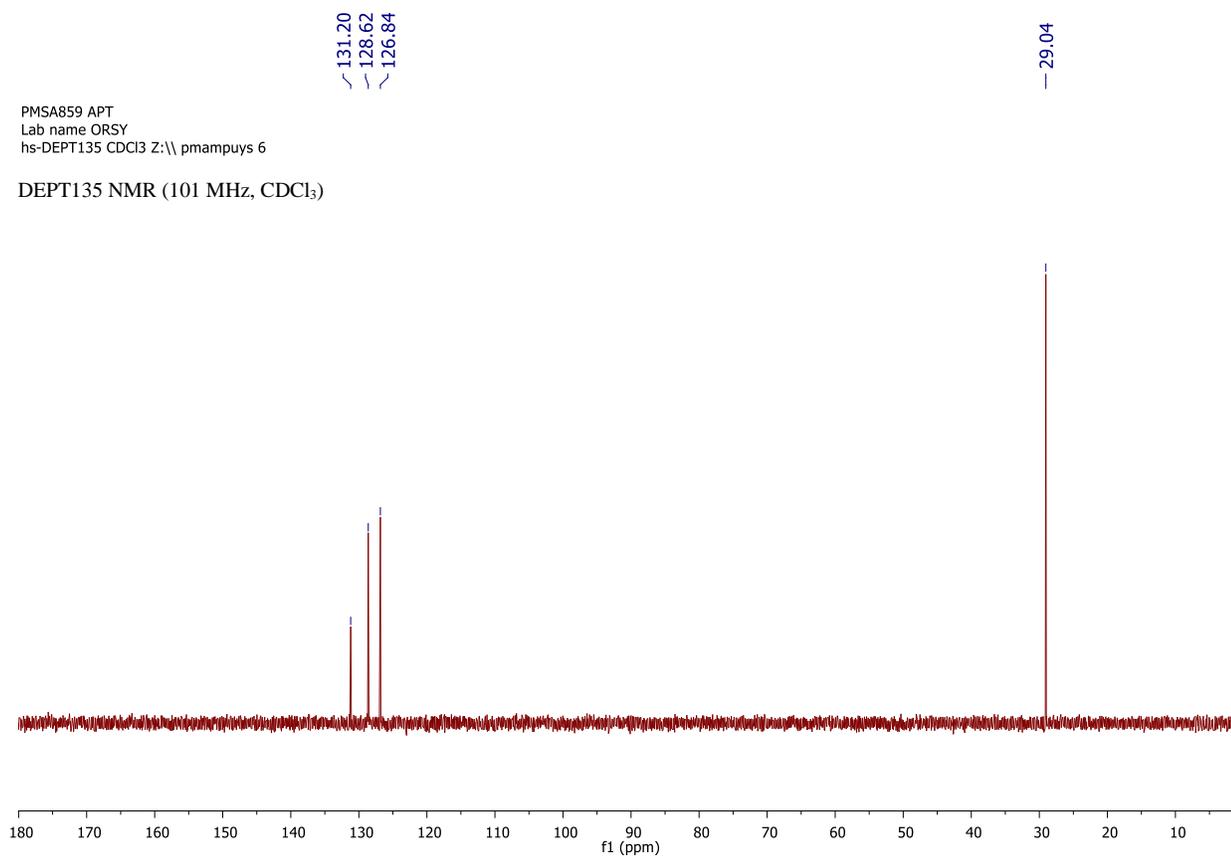
DEPT135 NMR (101 MHz, CDCl₃)



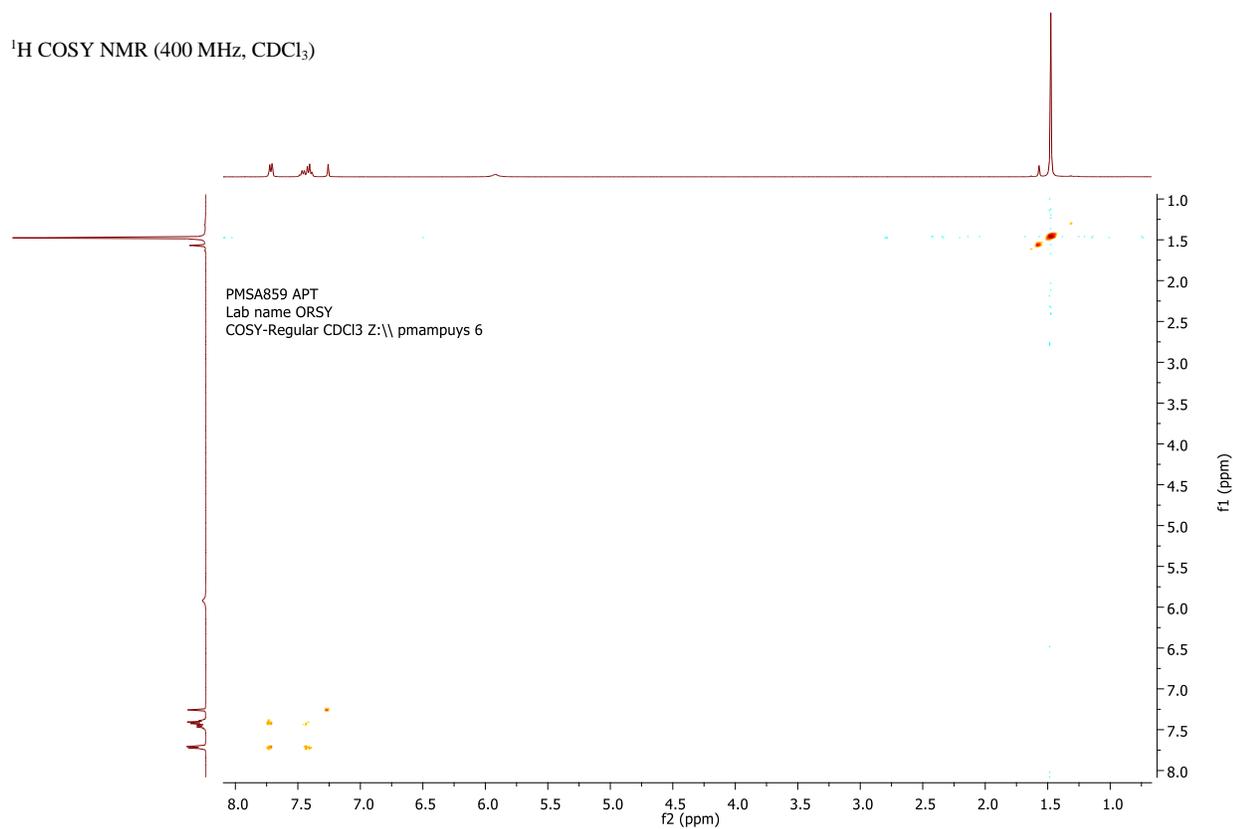
¹H COSY NMR (400 MHz, CDCl₃)

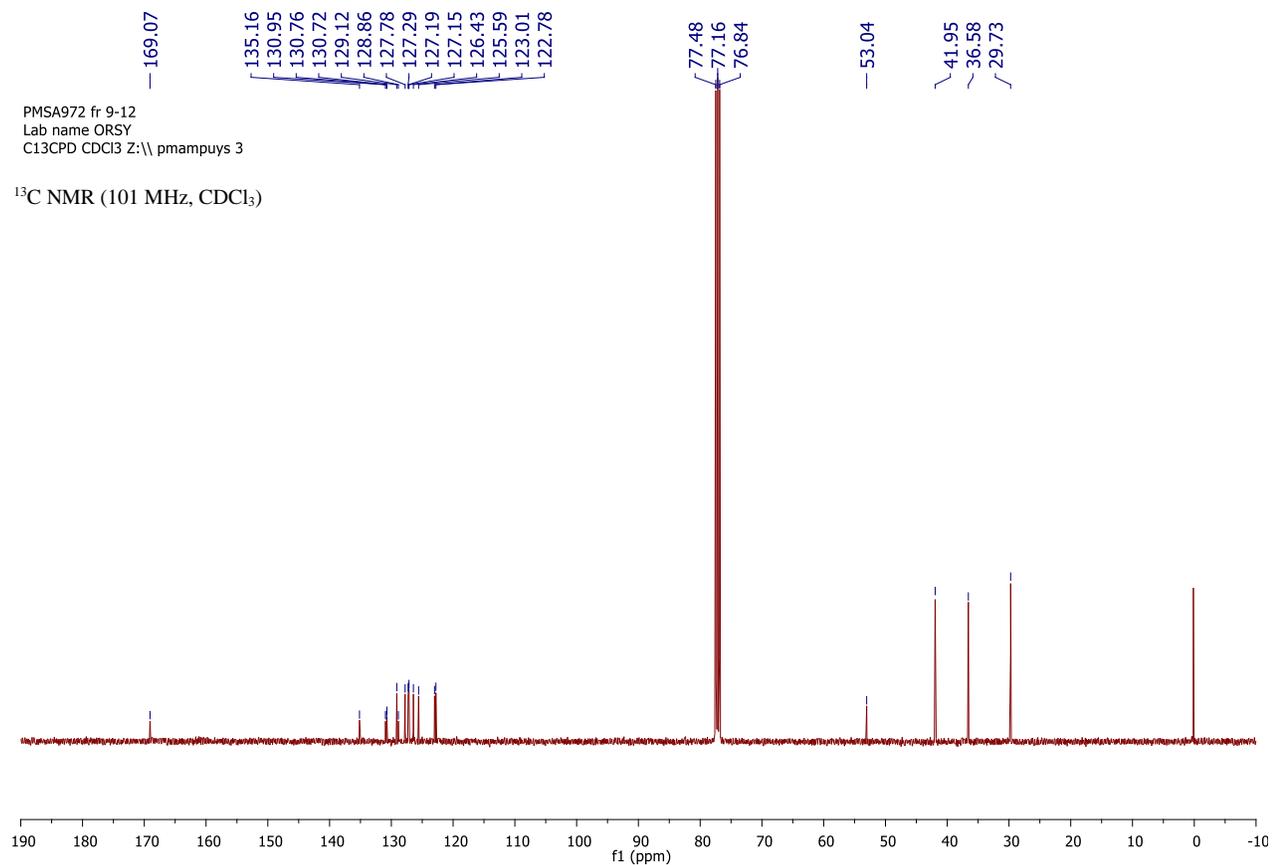
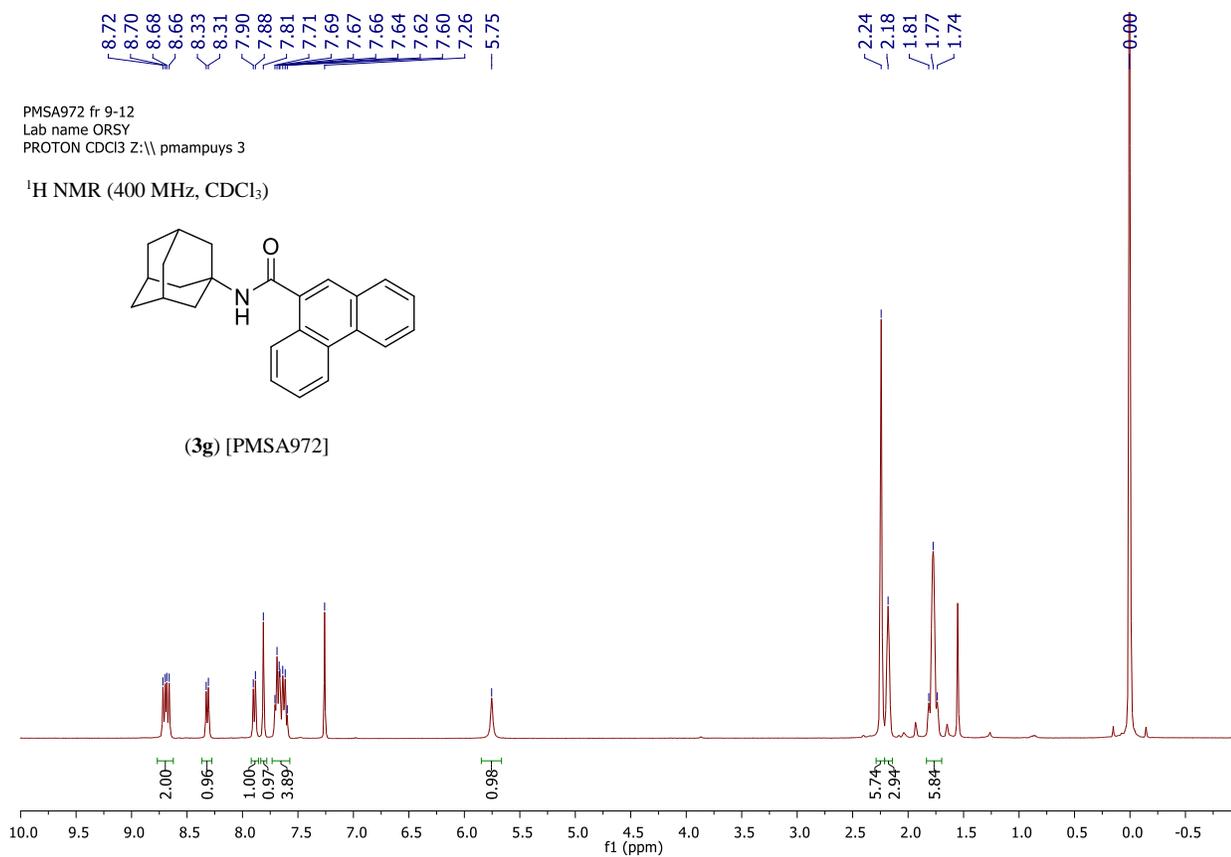




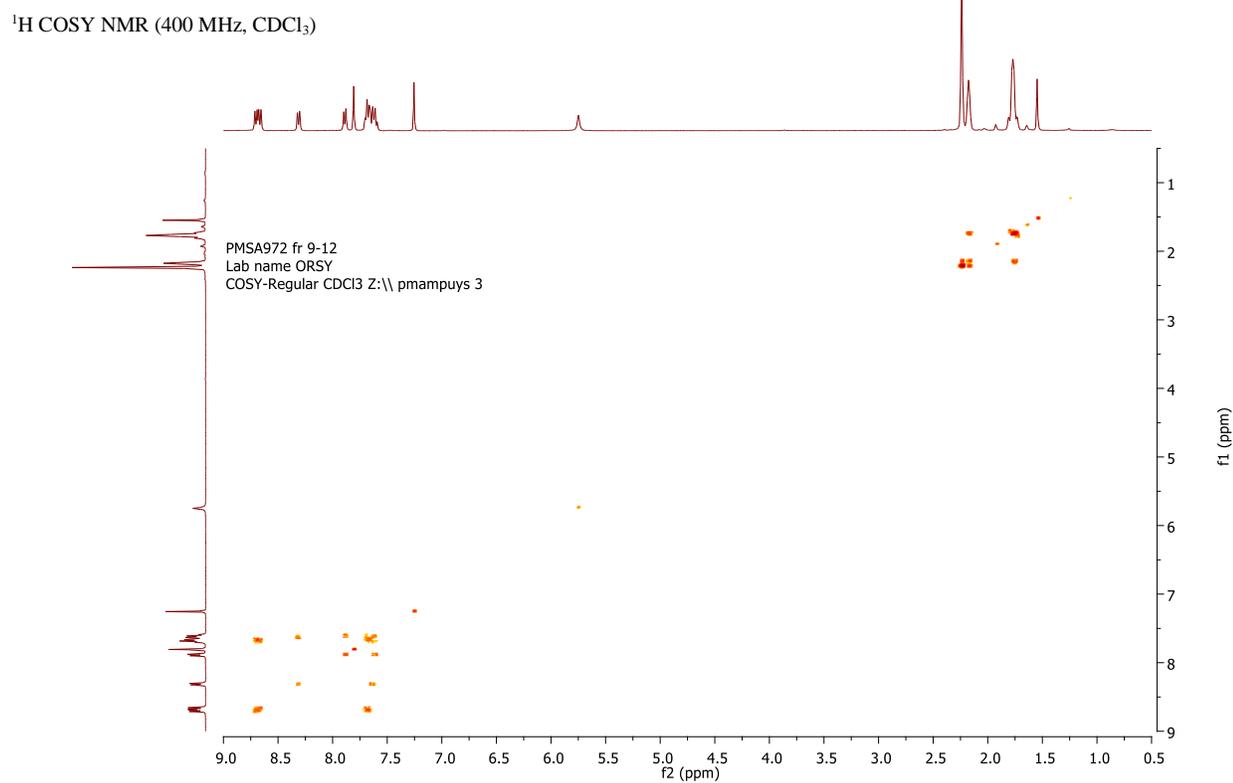
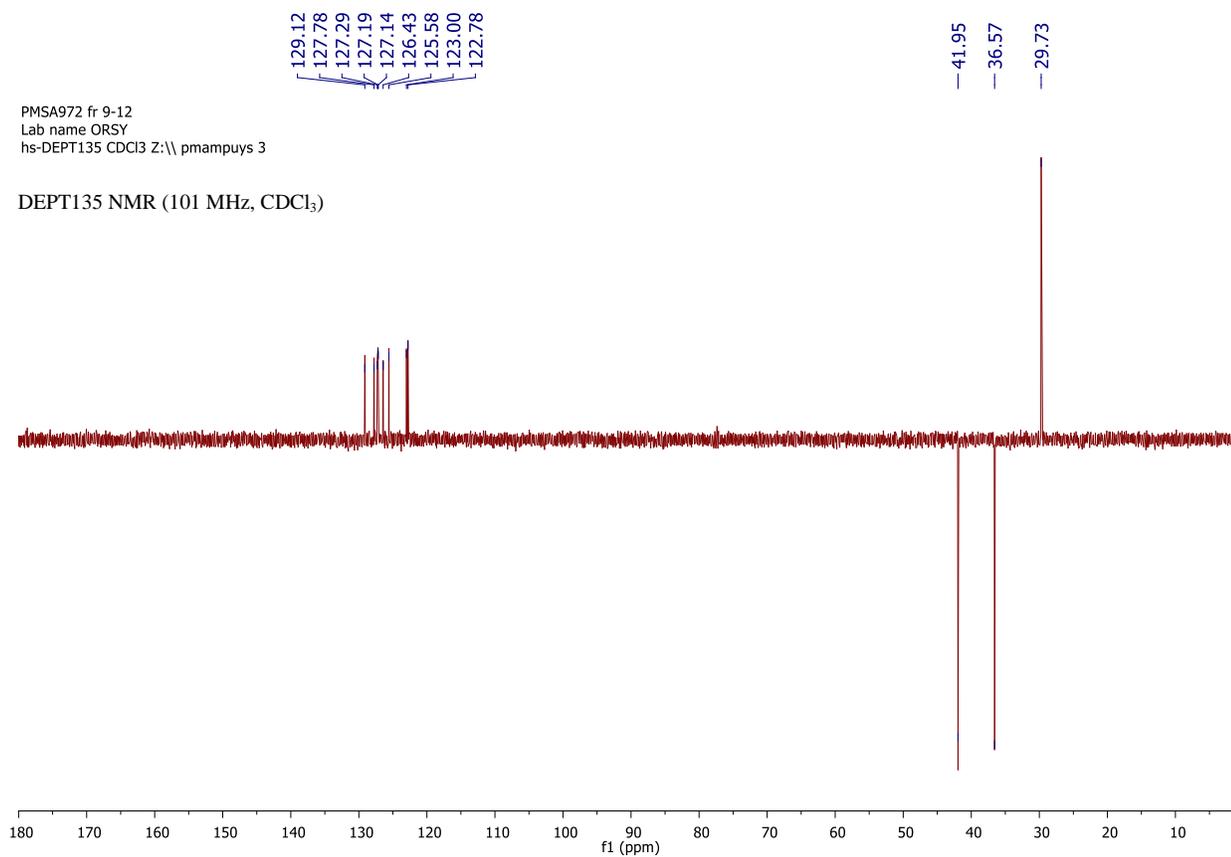


¹H COSY NMR (400 MHz, CDCl₃)

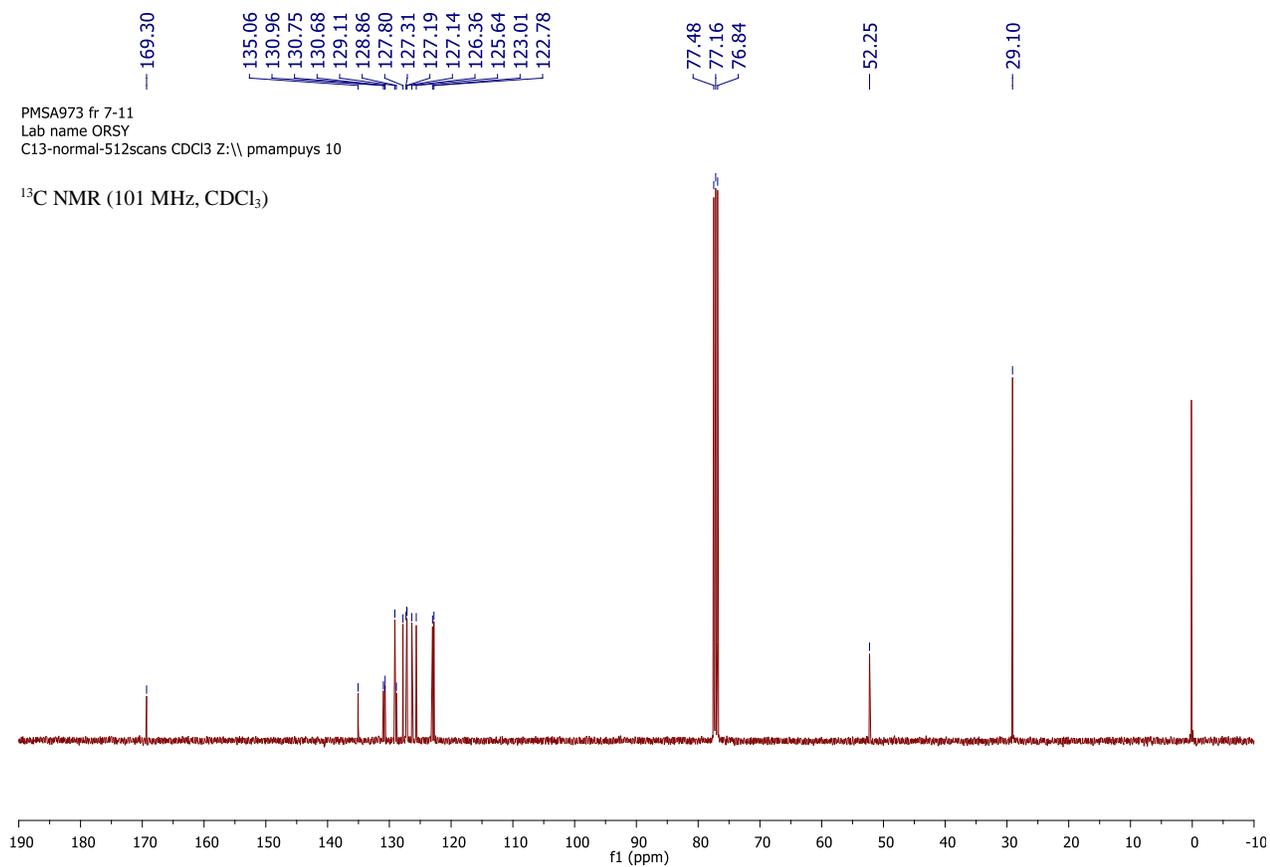
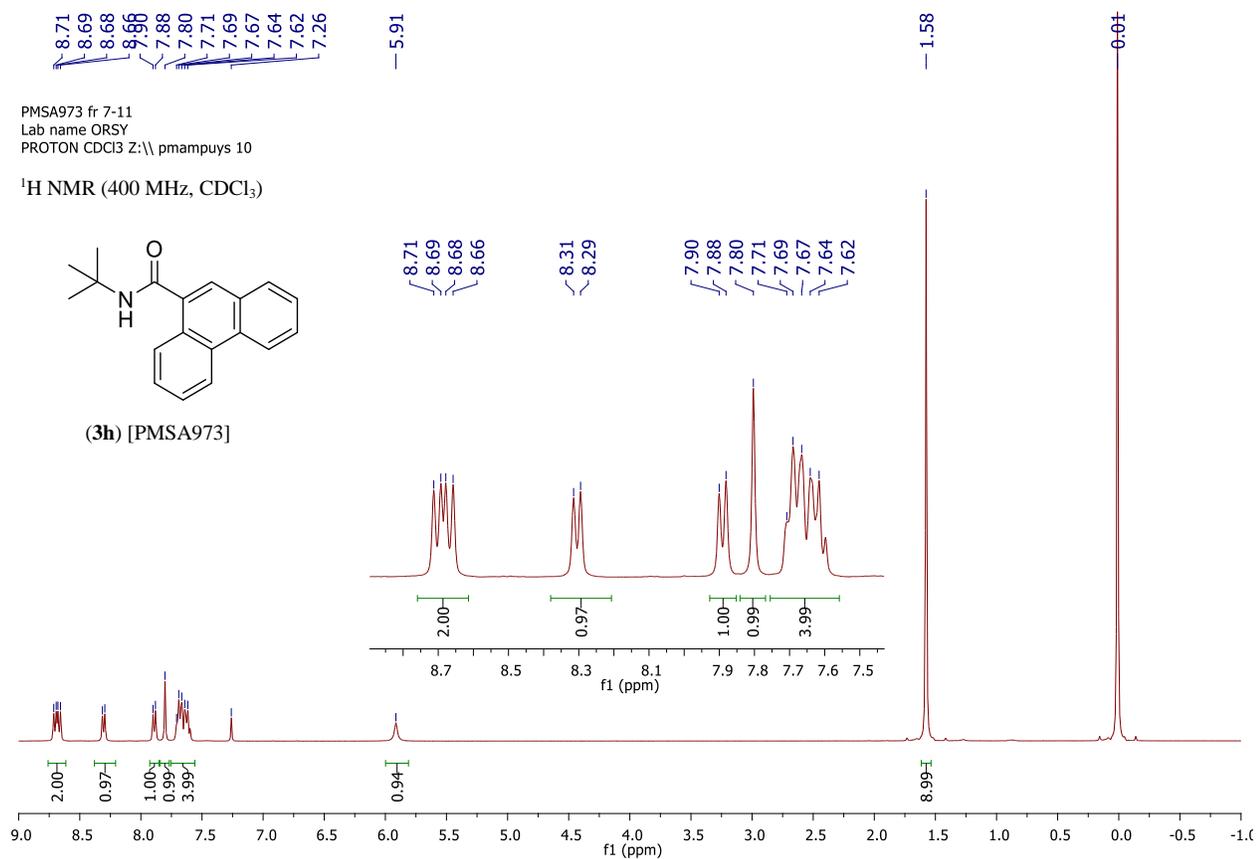




Copies of the NMR spectra



Copies of the NMR spectra

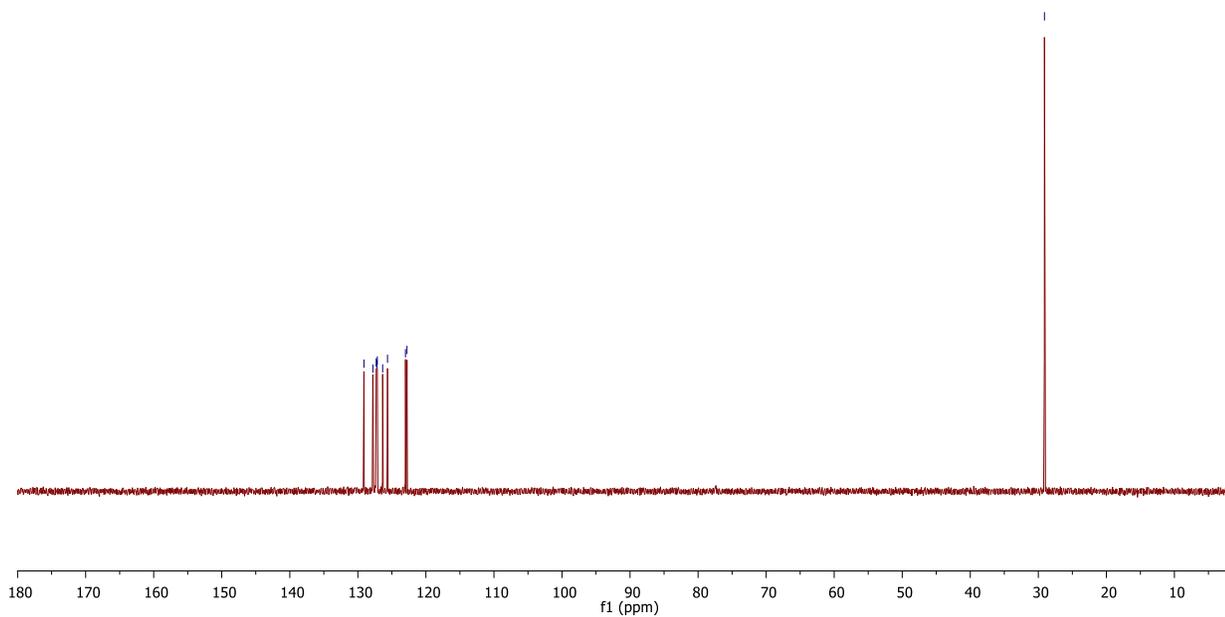


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127.30
127.19
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126.36
125.64
123.00
122.78

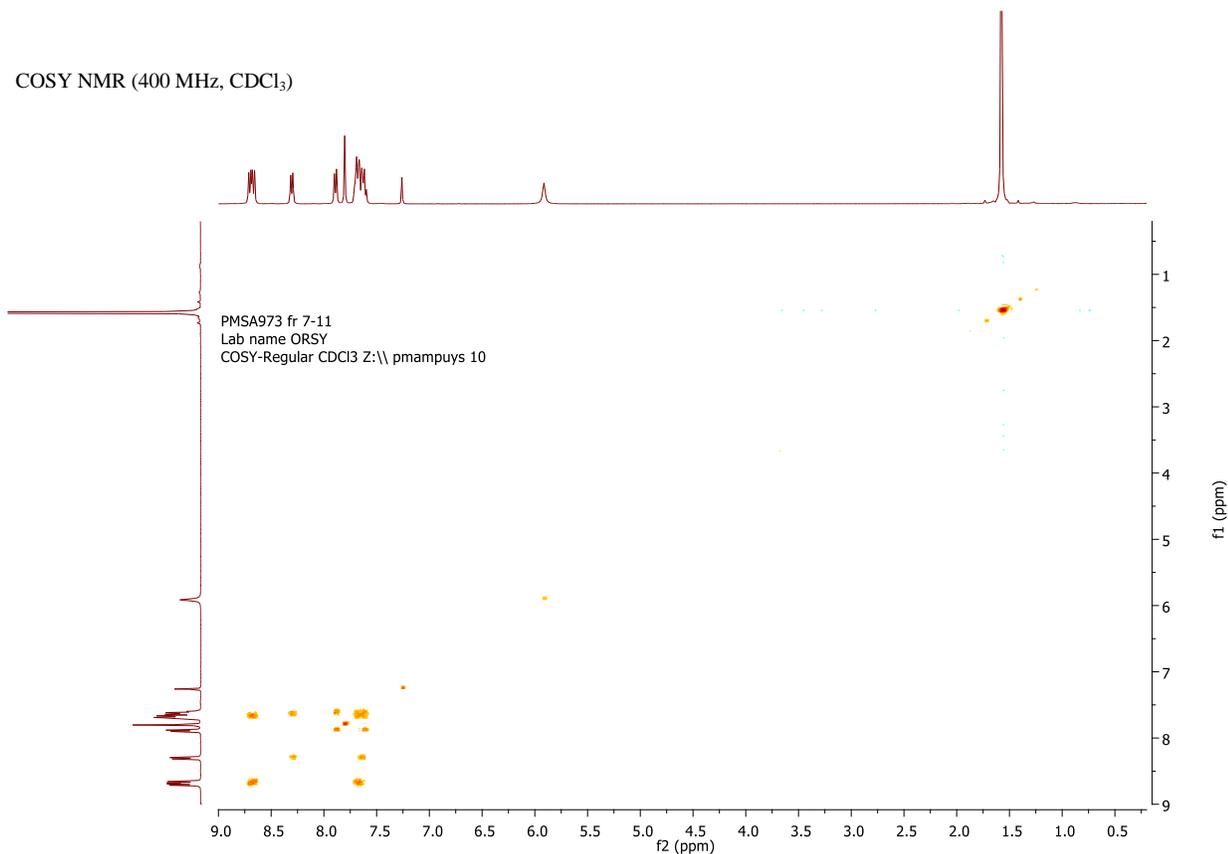
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PMSA973 fr 7-11
Lab name ORSY
hs-DEPT135 CDCl₃ Z:\\ pmampuys 10

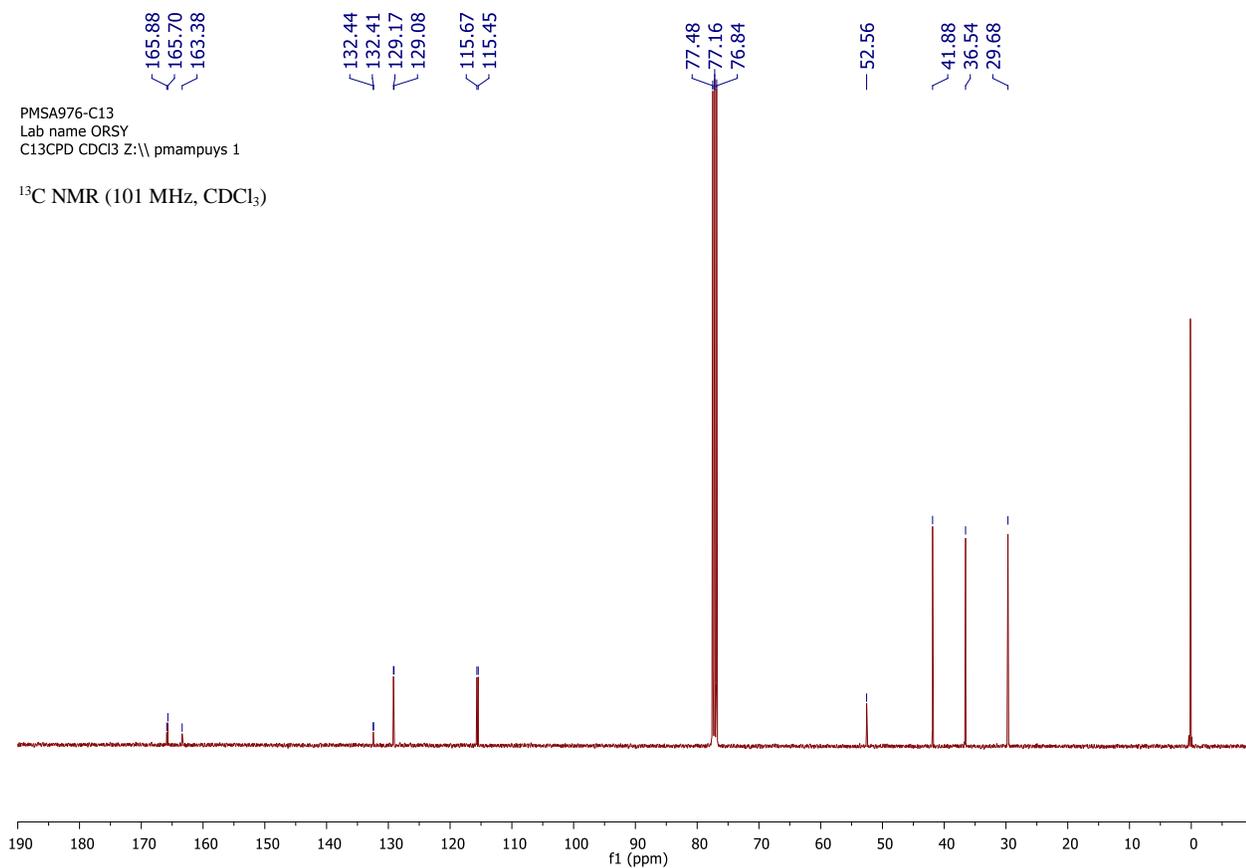
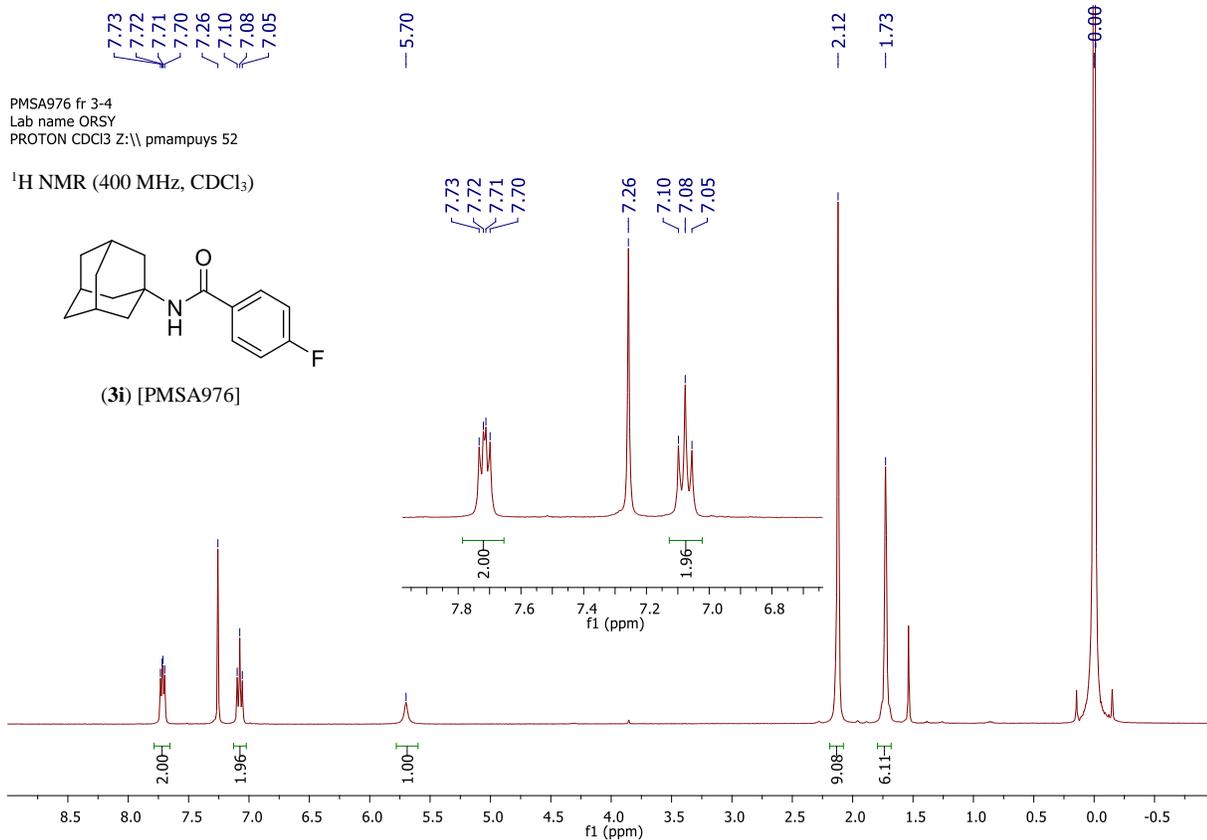
DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)



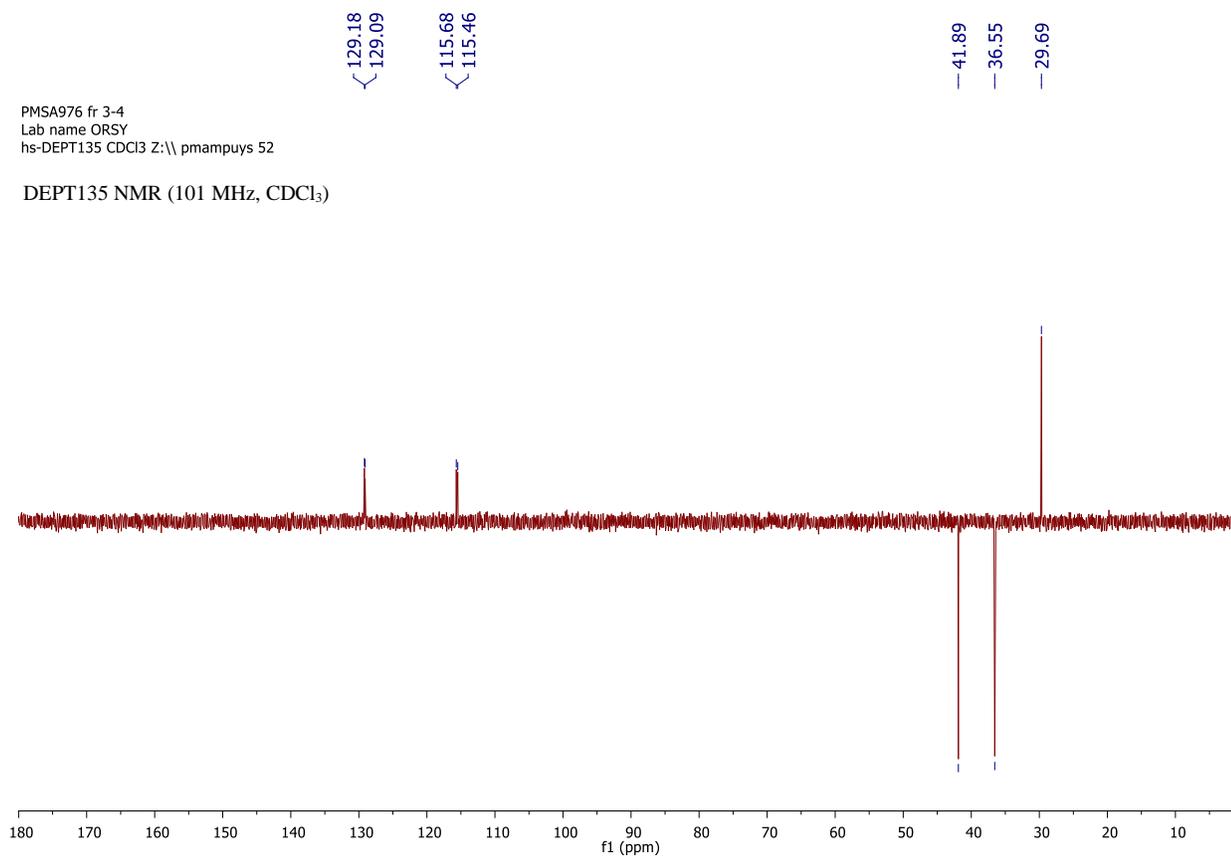
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Lab name ORSY
COSY-Regular CDCl₃ Z:\\ pmampuys 10



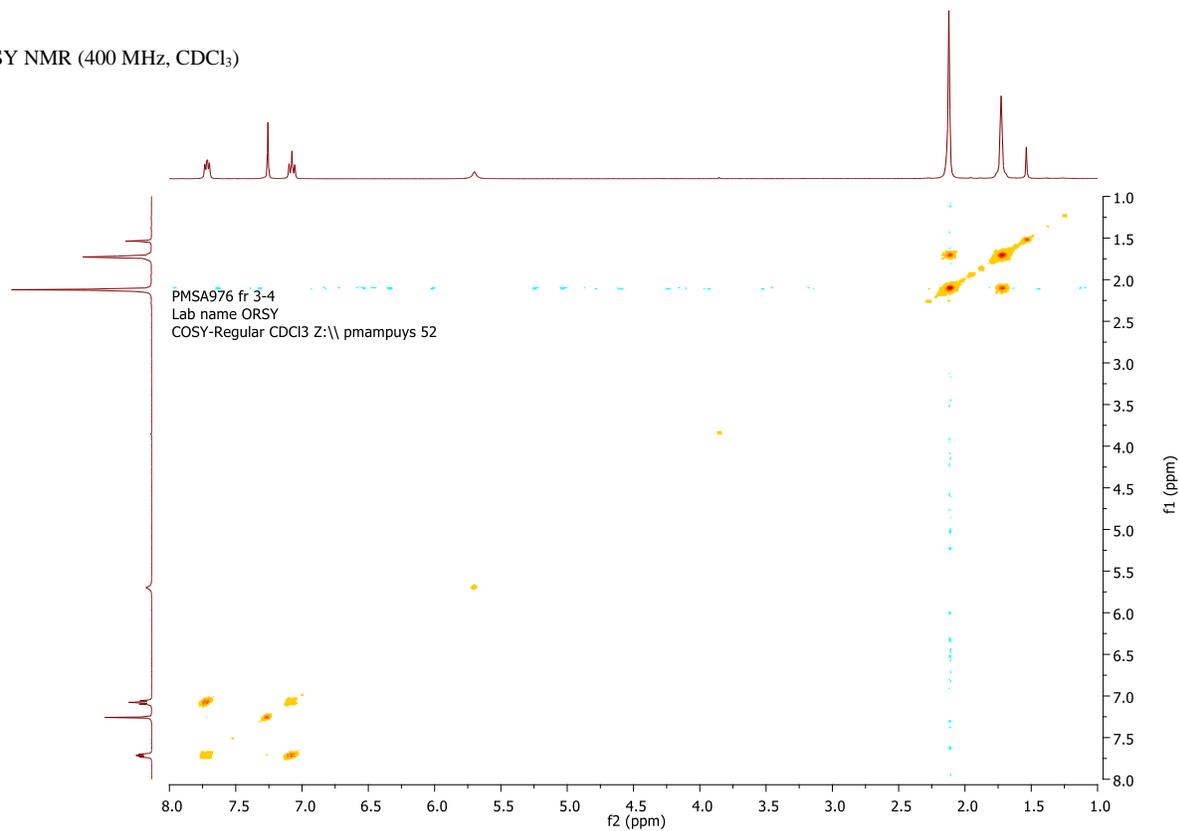
Copies of the NMR spectra

PMSA976 fr 3-4
Lab name ORSY
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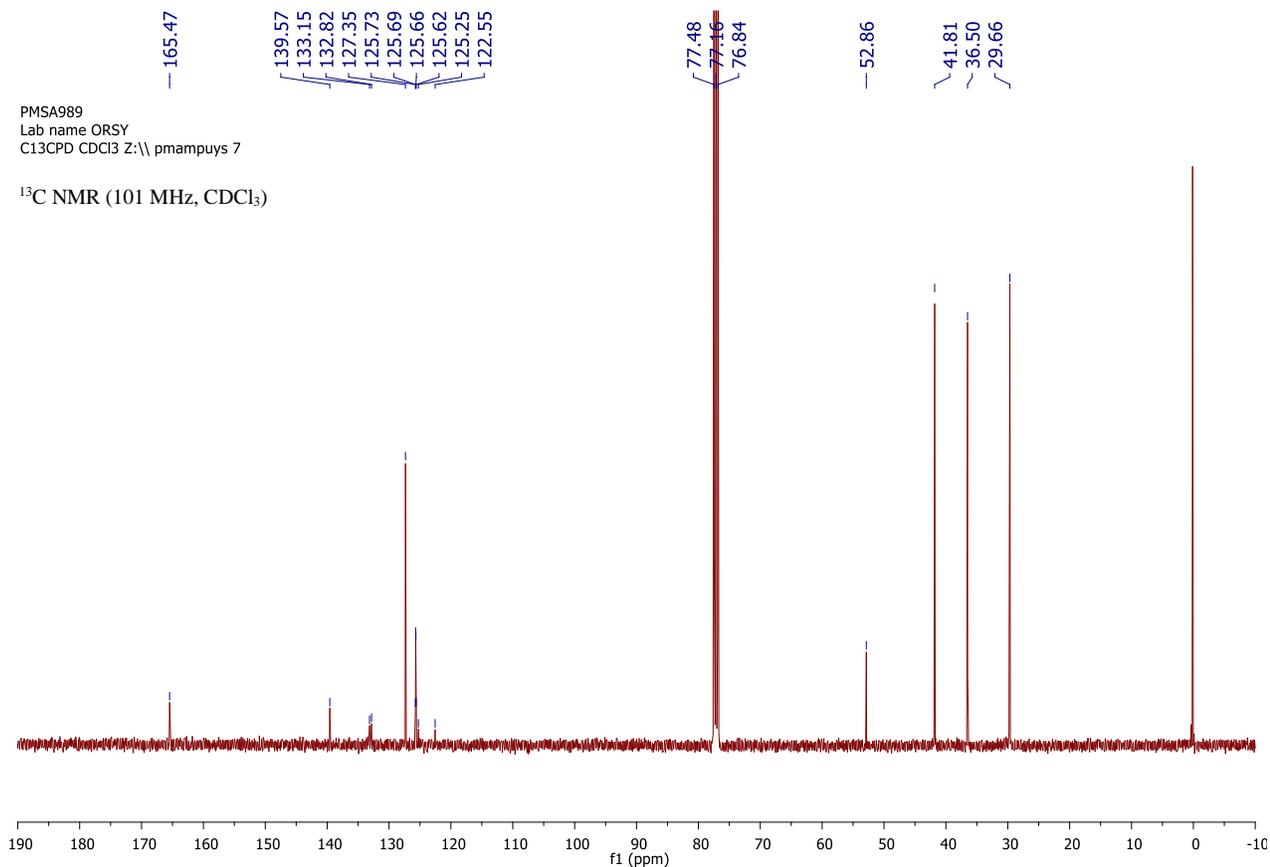
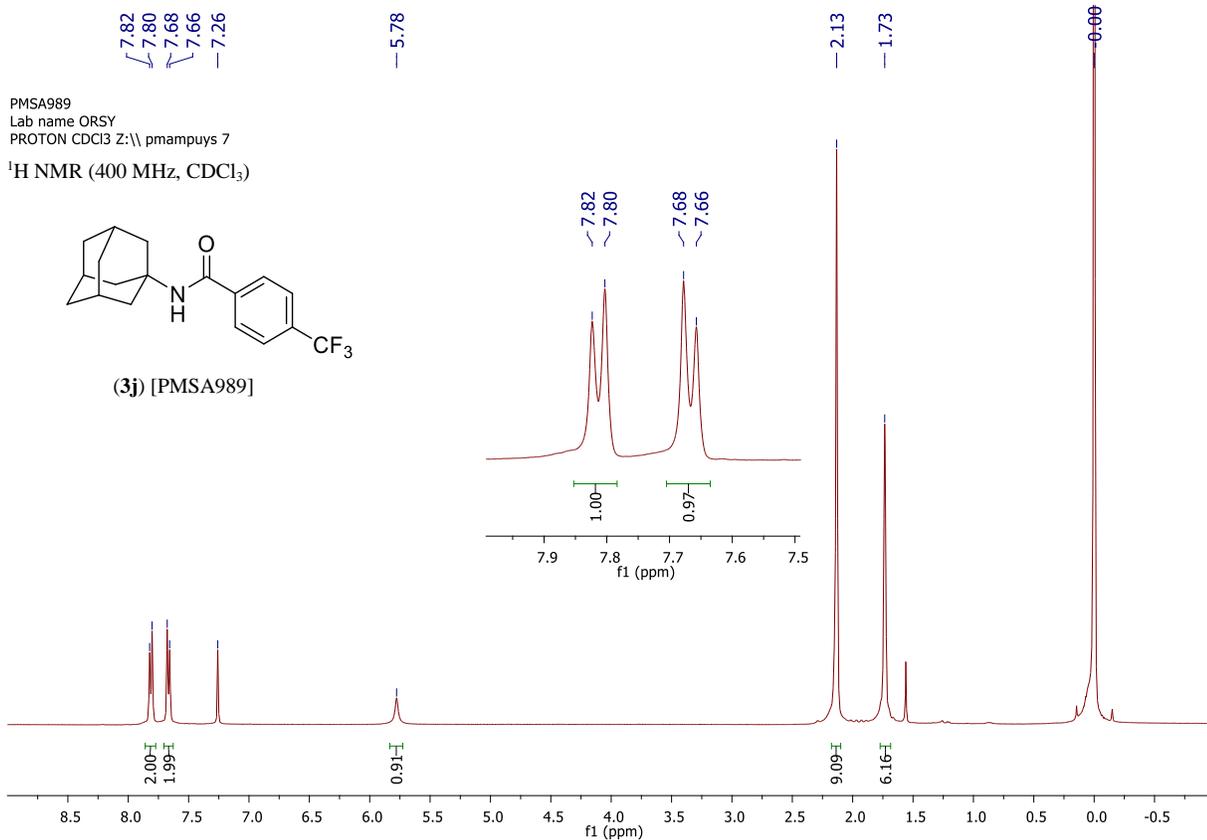
DEPT135 NMR (101 MHz, CDCl₃)



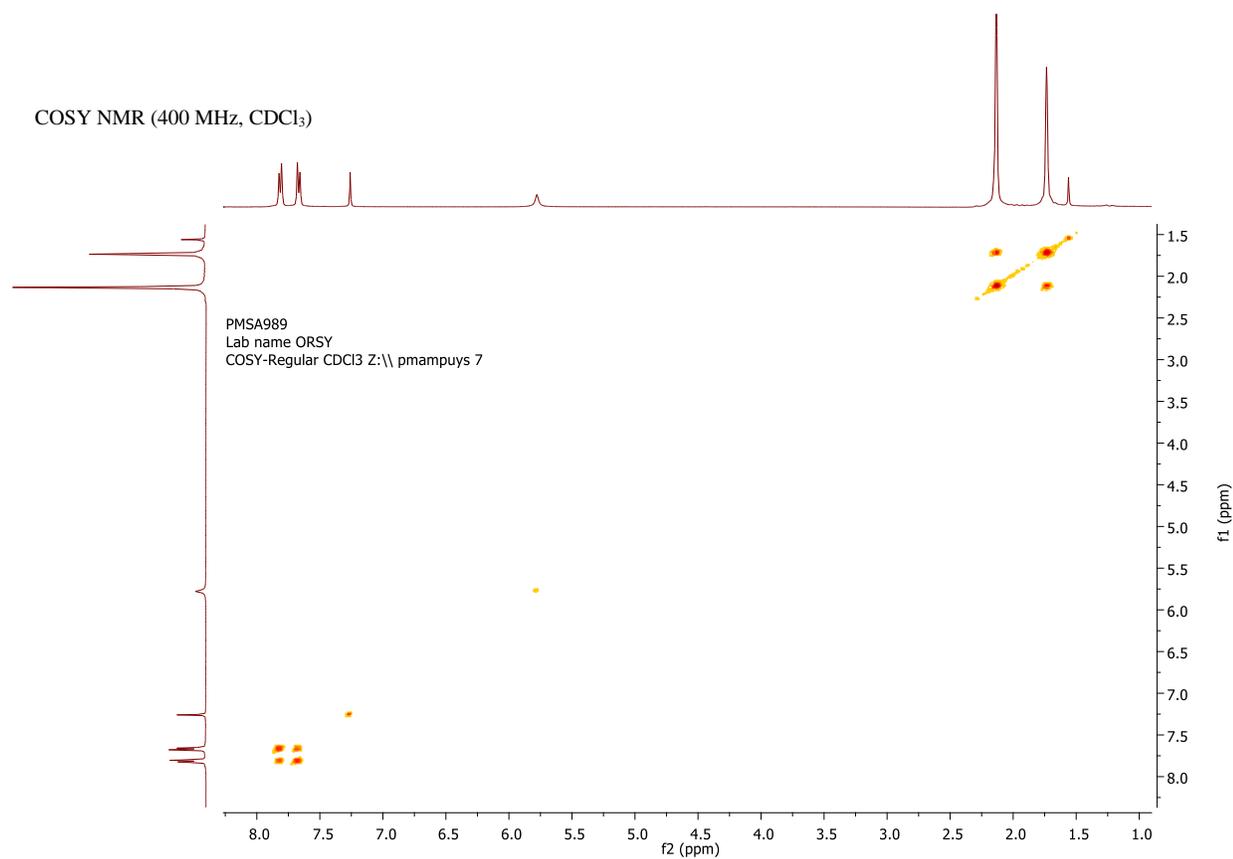
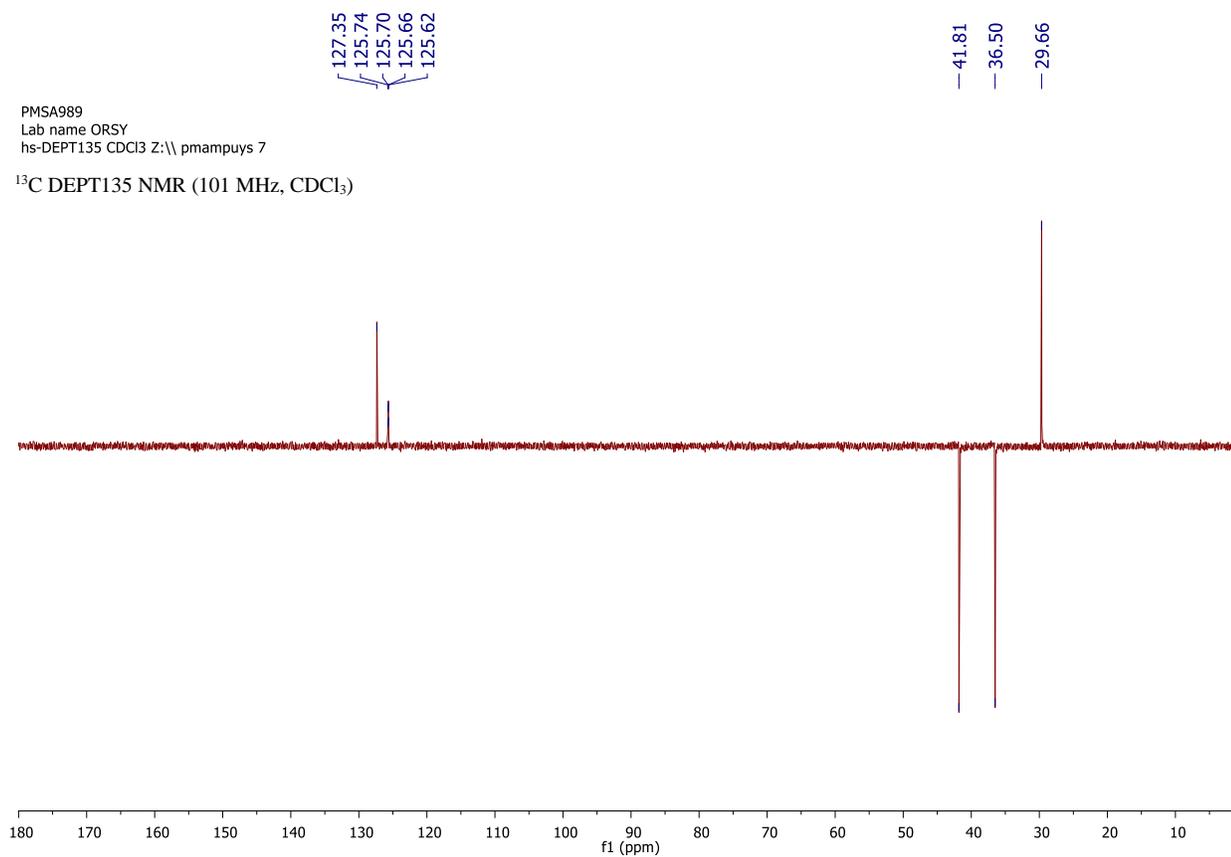
COSY NMR (400 MHz, CDCl₃)

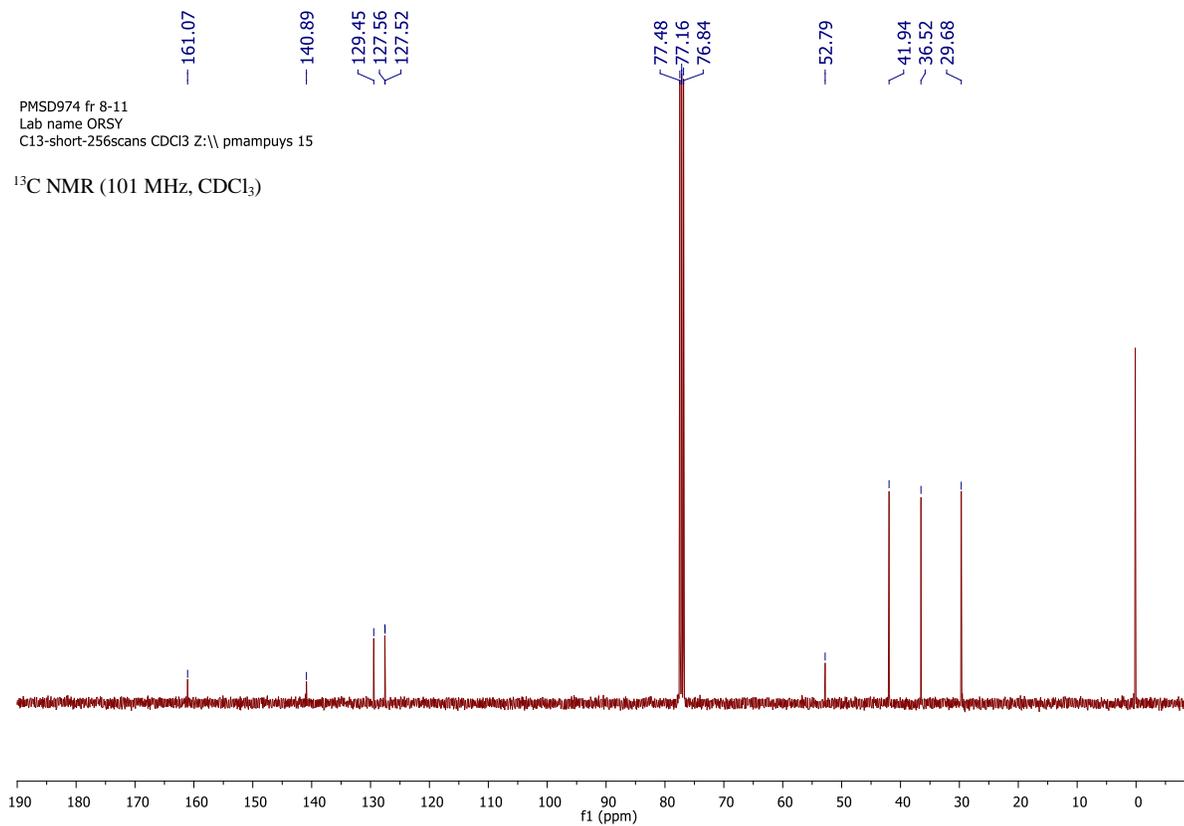
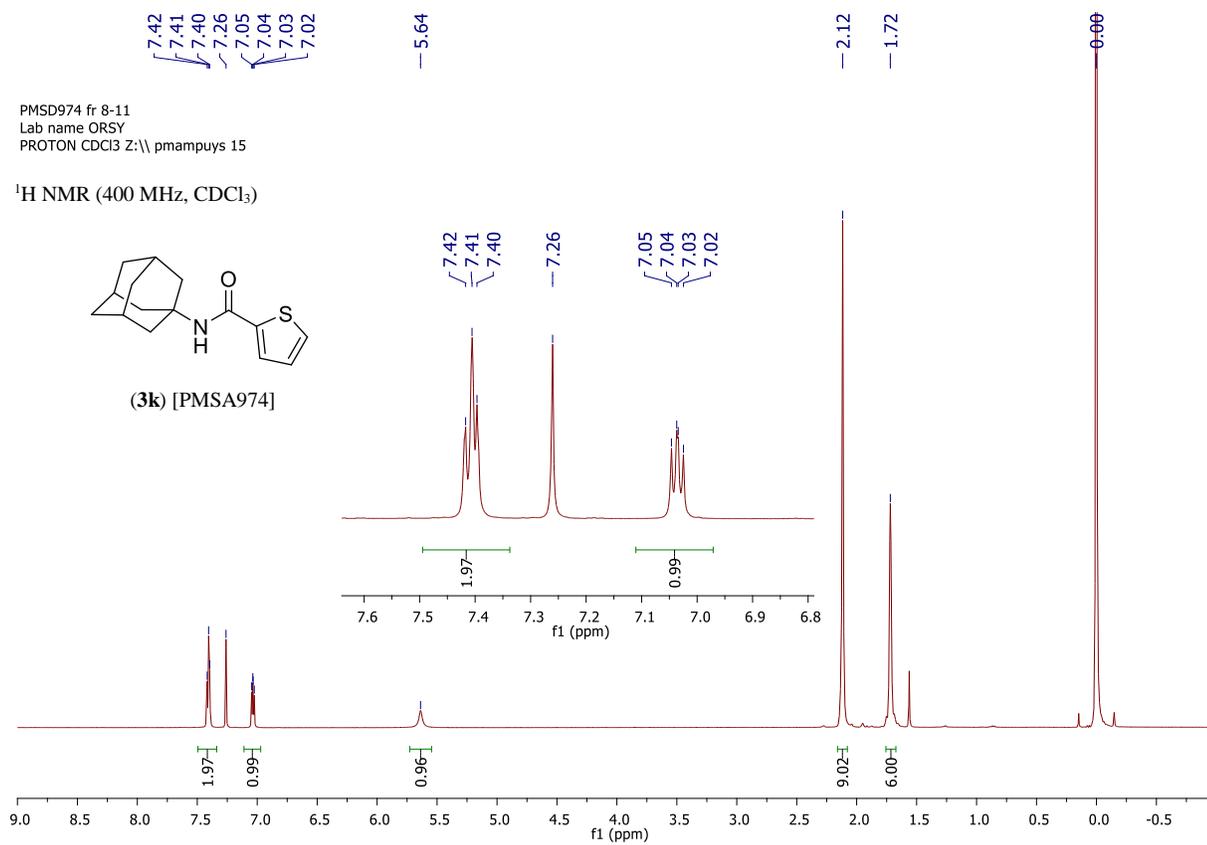


Copies of the NMR spectra



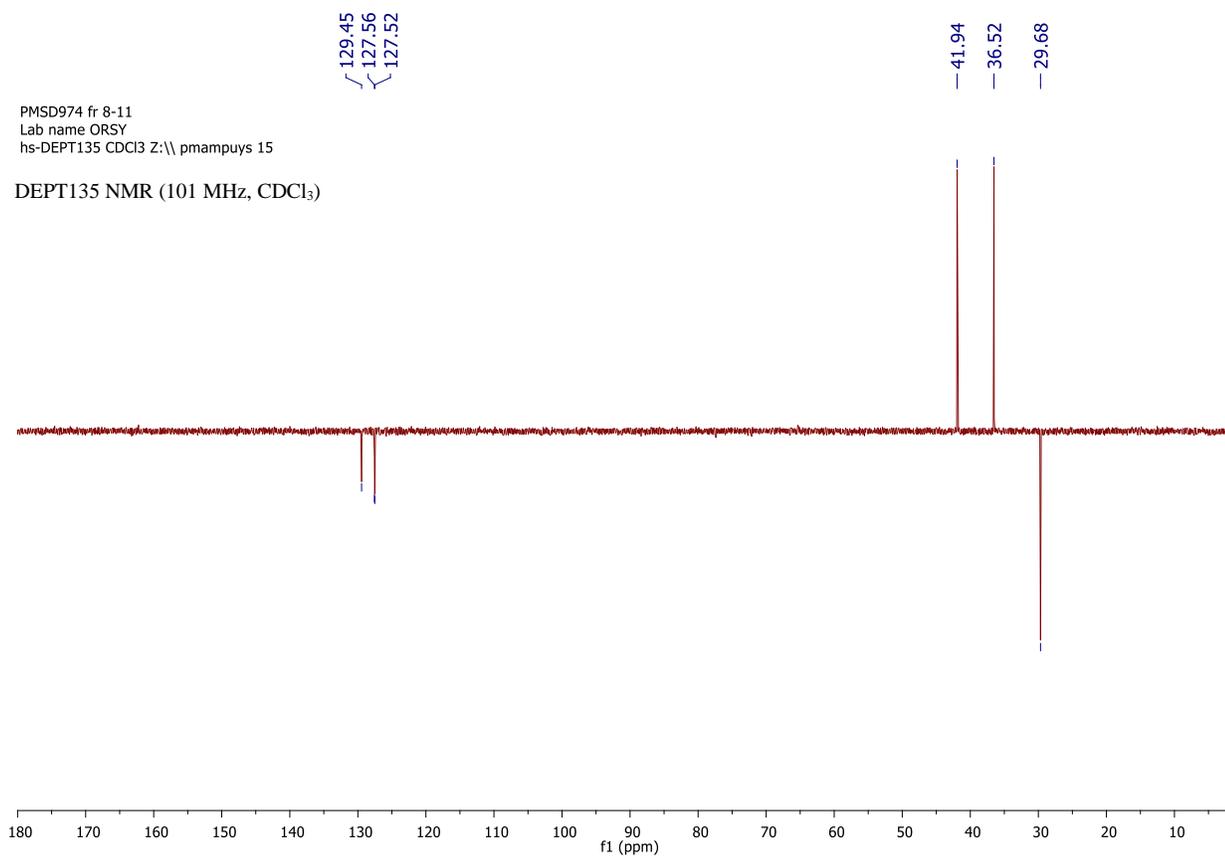
Copies of the NMR spectra



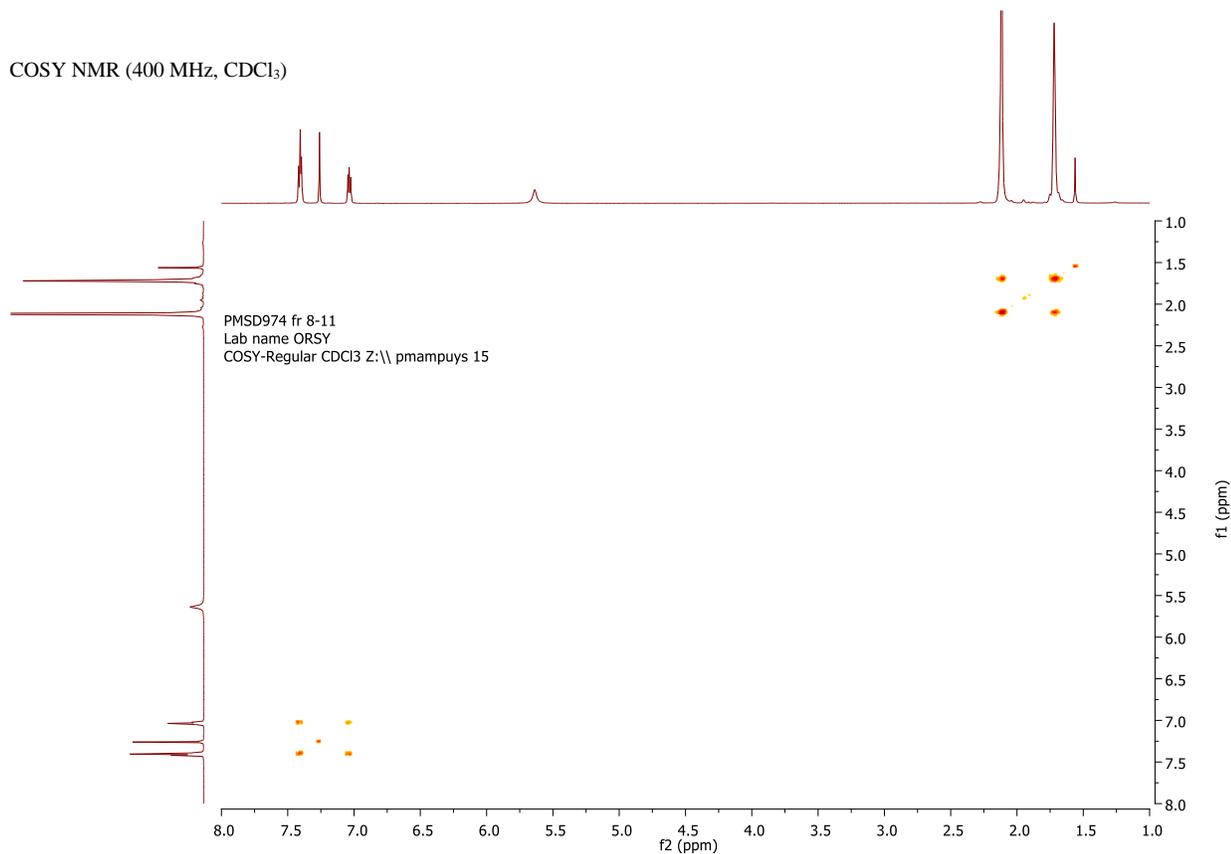


PMSD974 fr 8-11
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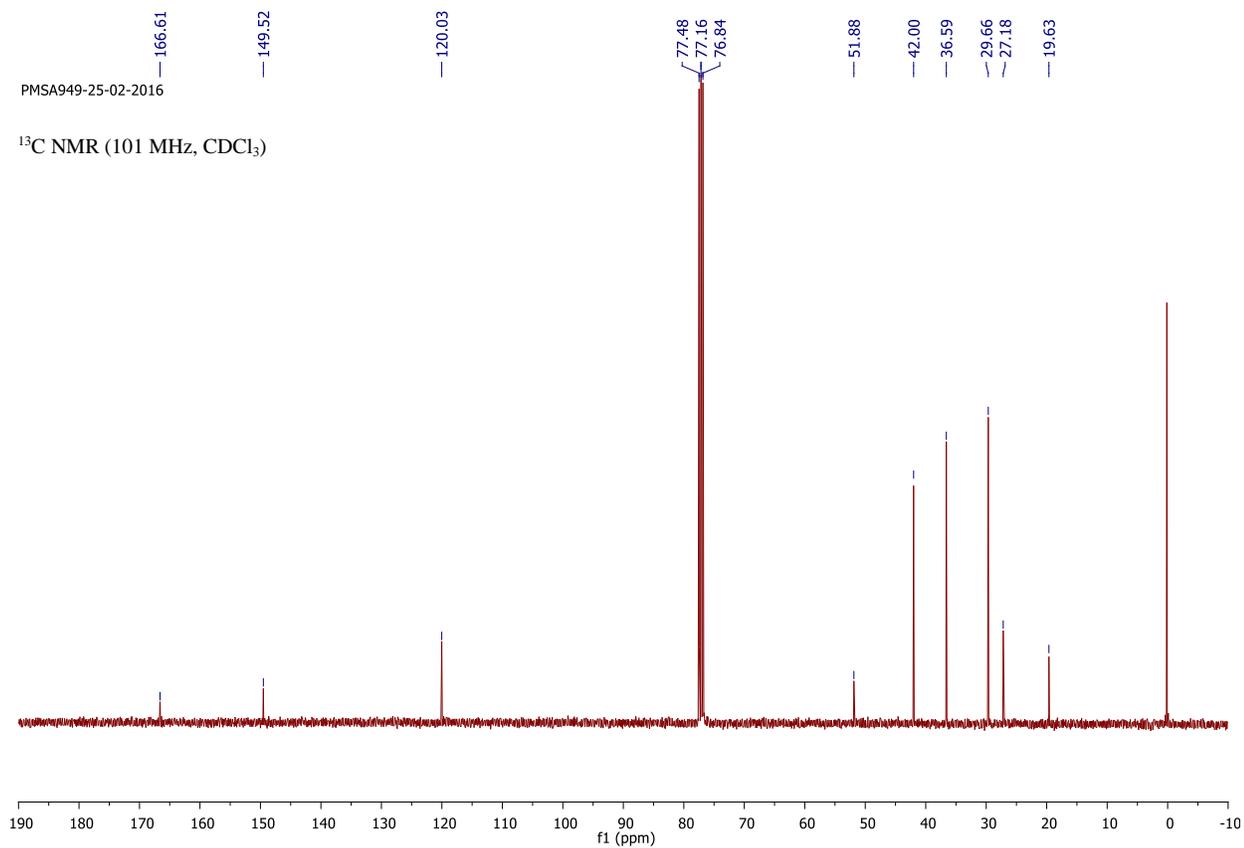
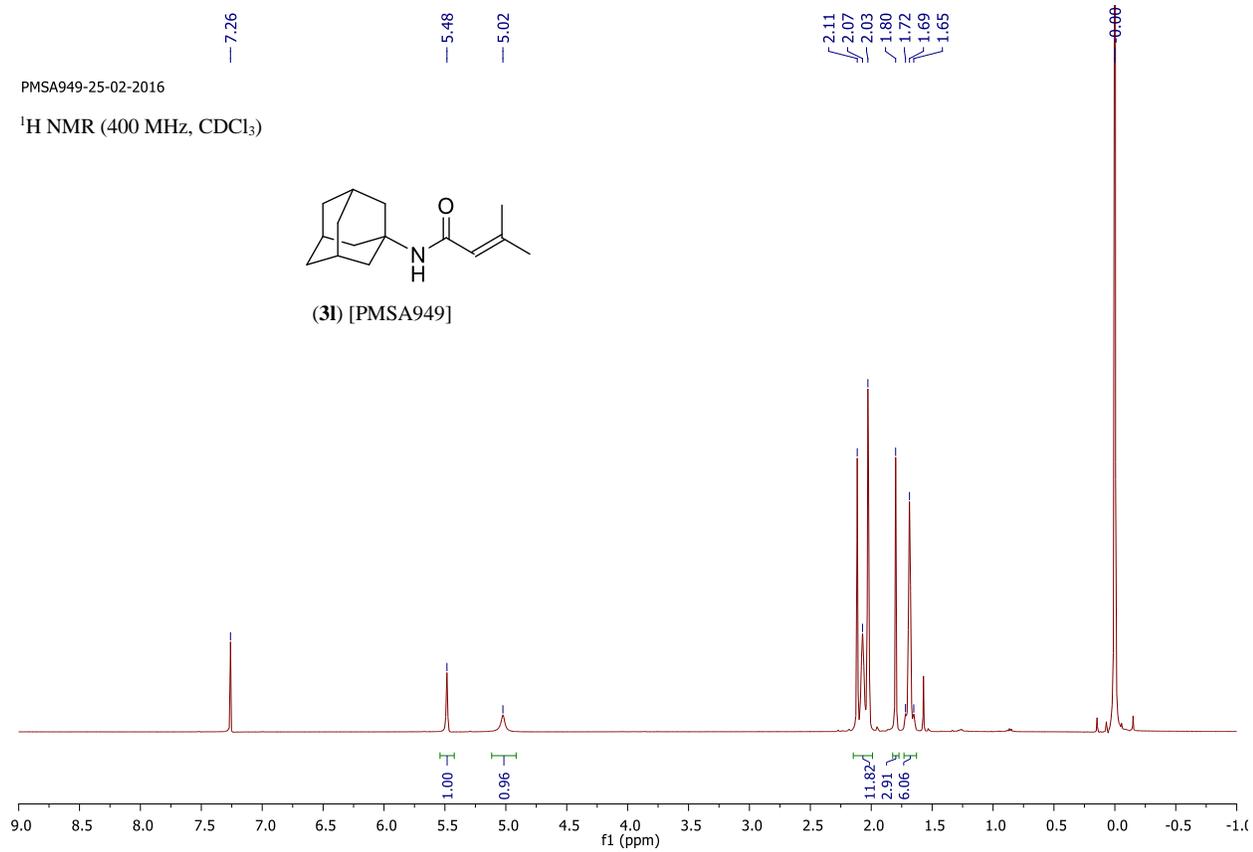
DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)



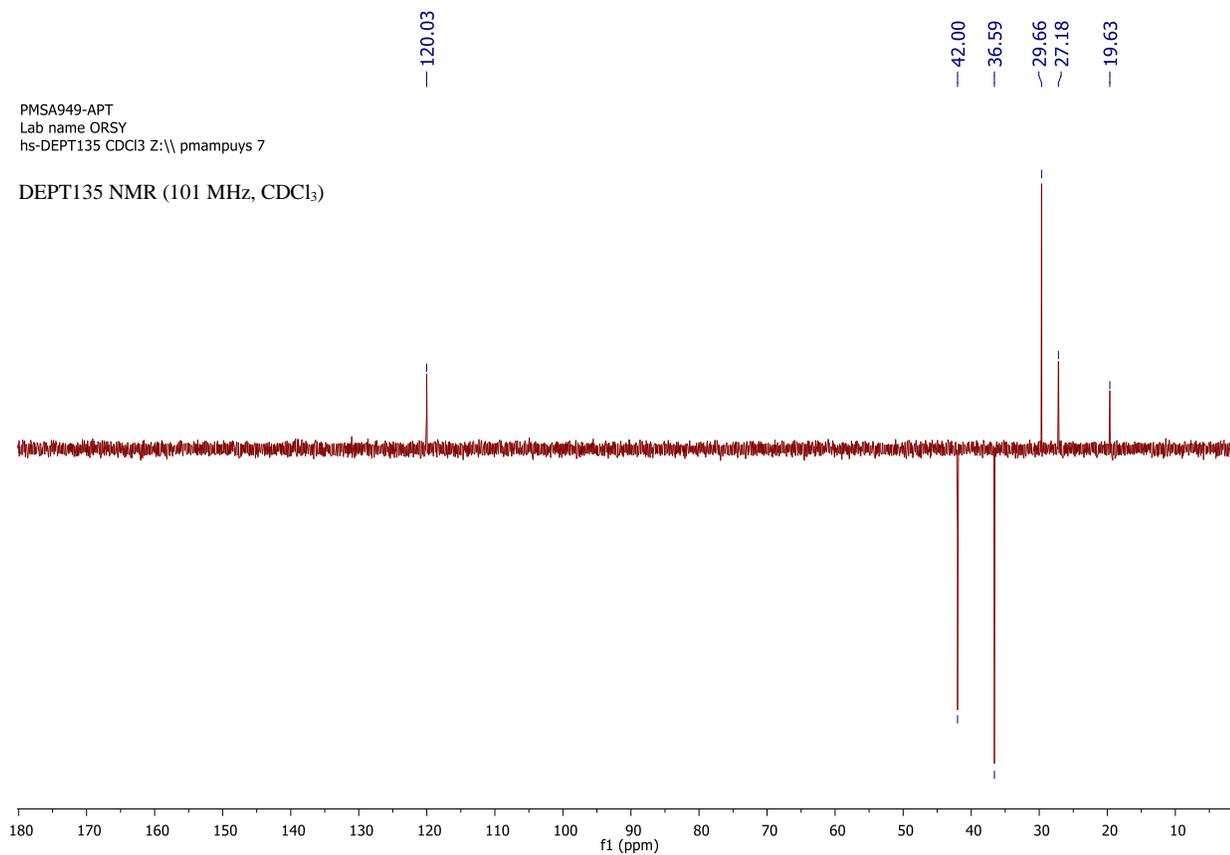
Copies of the NMR spectra



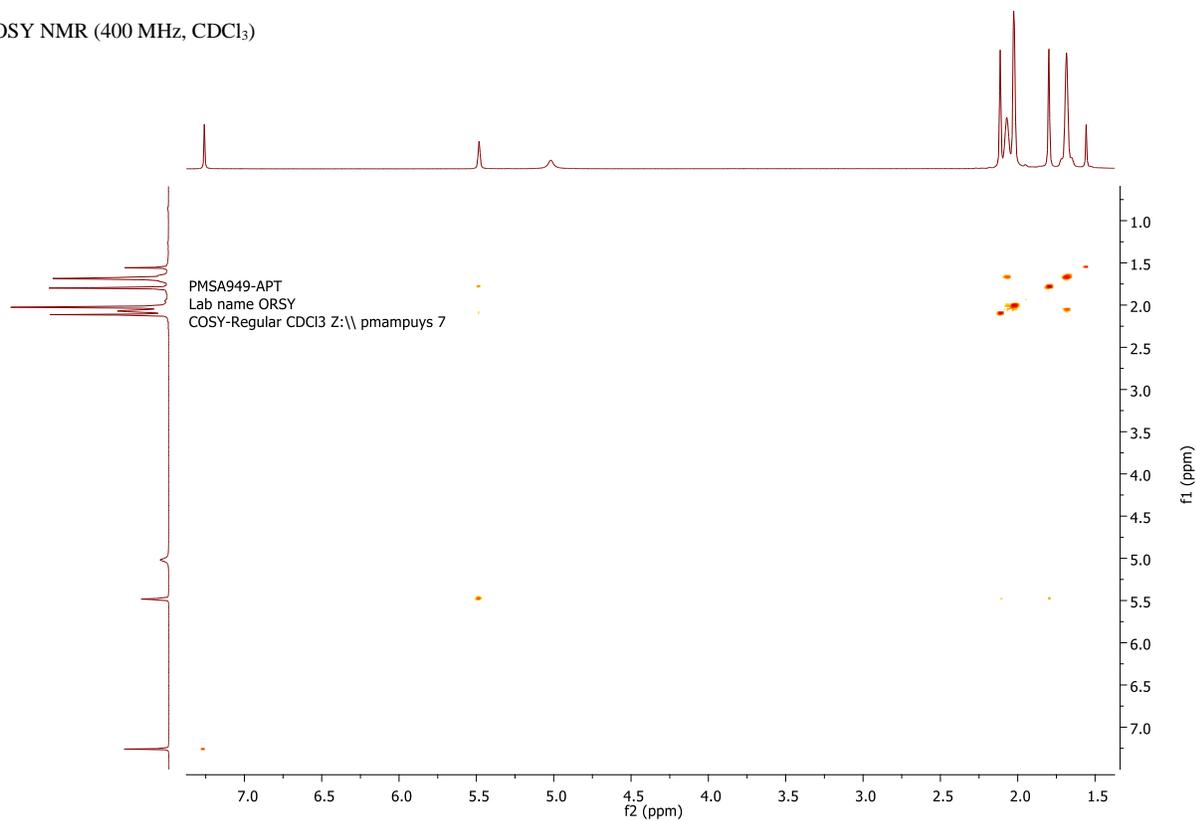
Copies of the NMR spectra

PMSA949-APT
Lab name ORSY
hs-DEPT135 CDCl₃ Z:\\ pmampuys 7

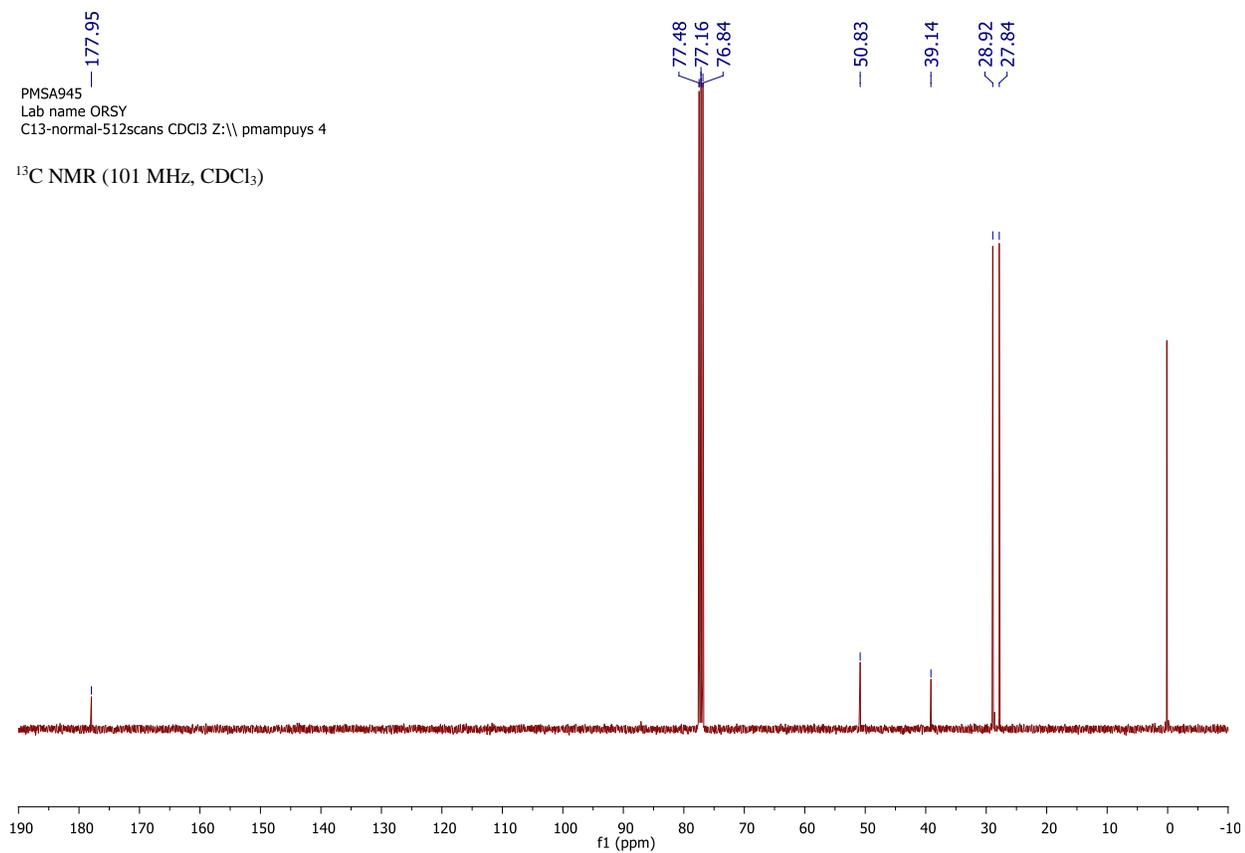
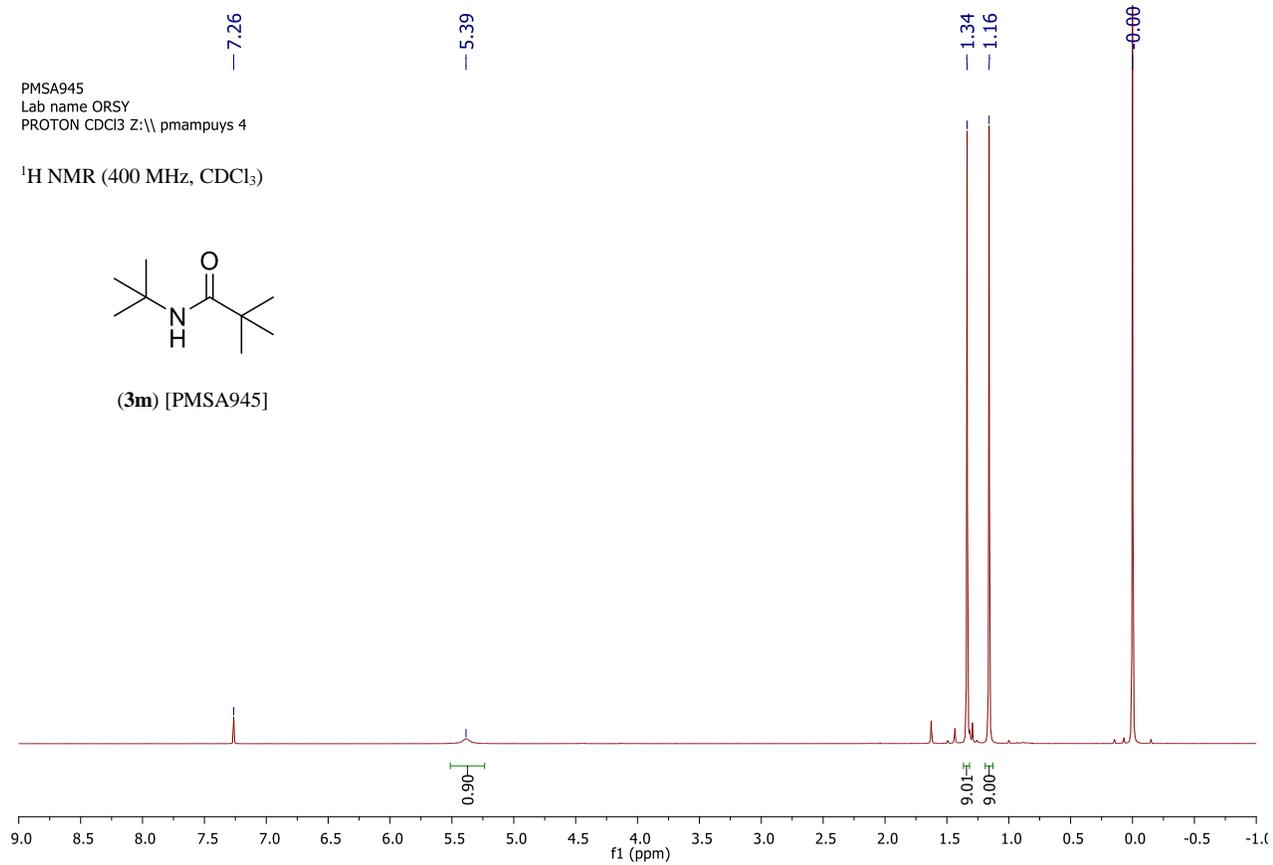
DEPT135 NMR (101 MHz, CDCl₃)



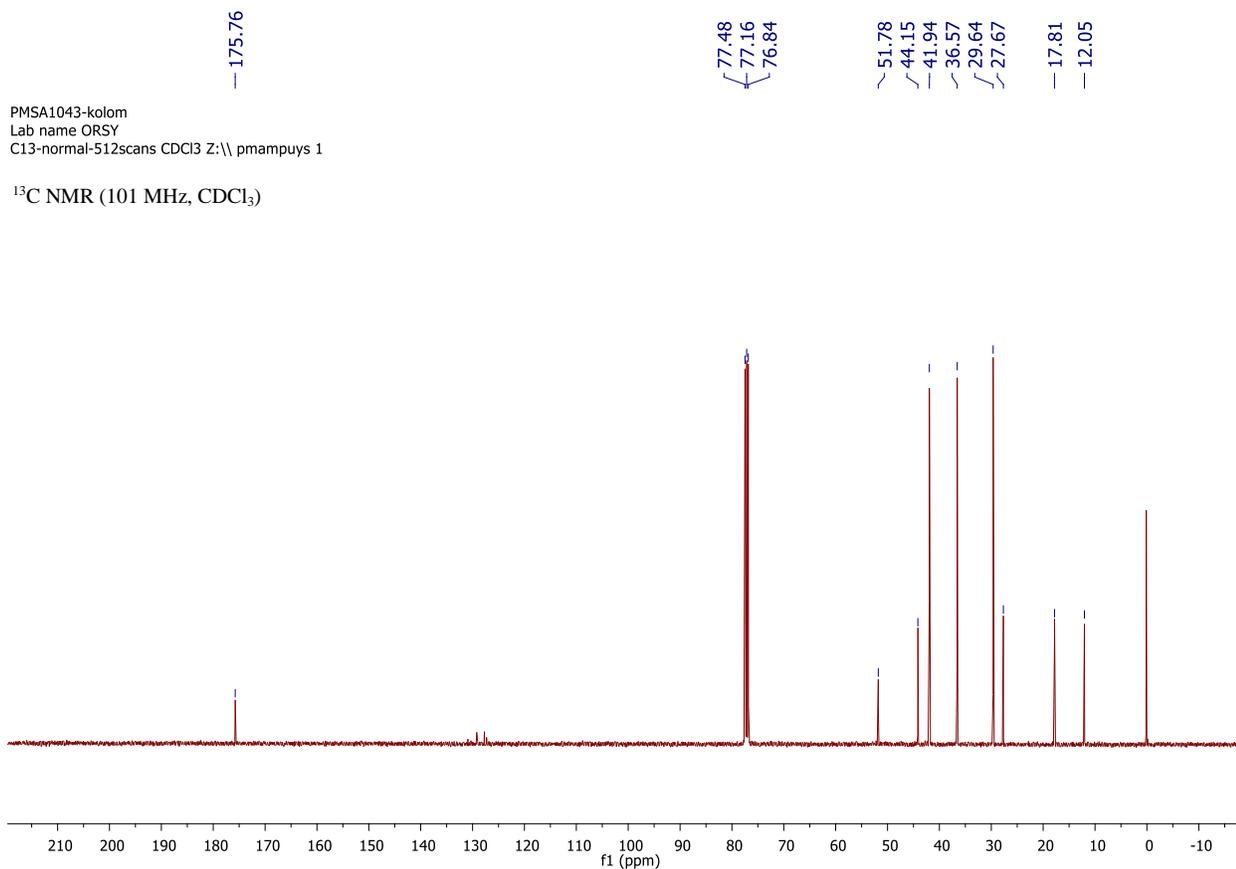
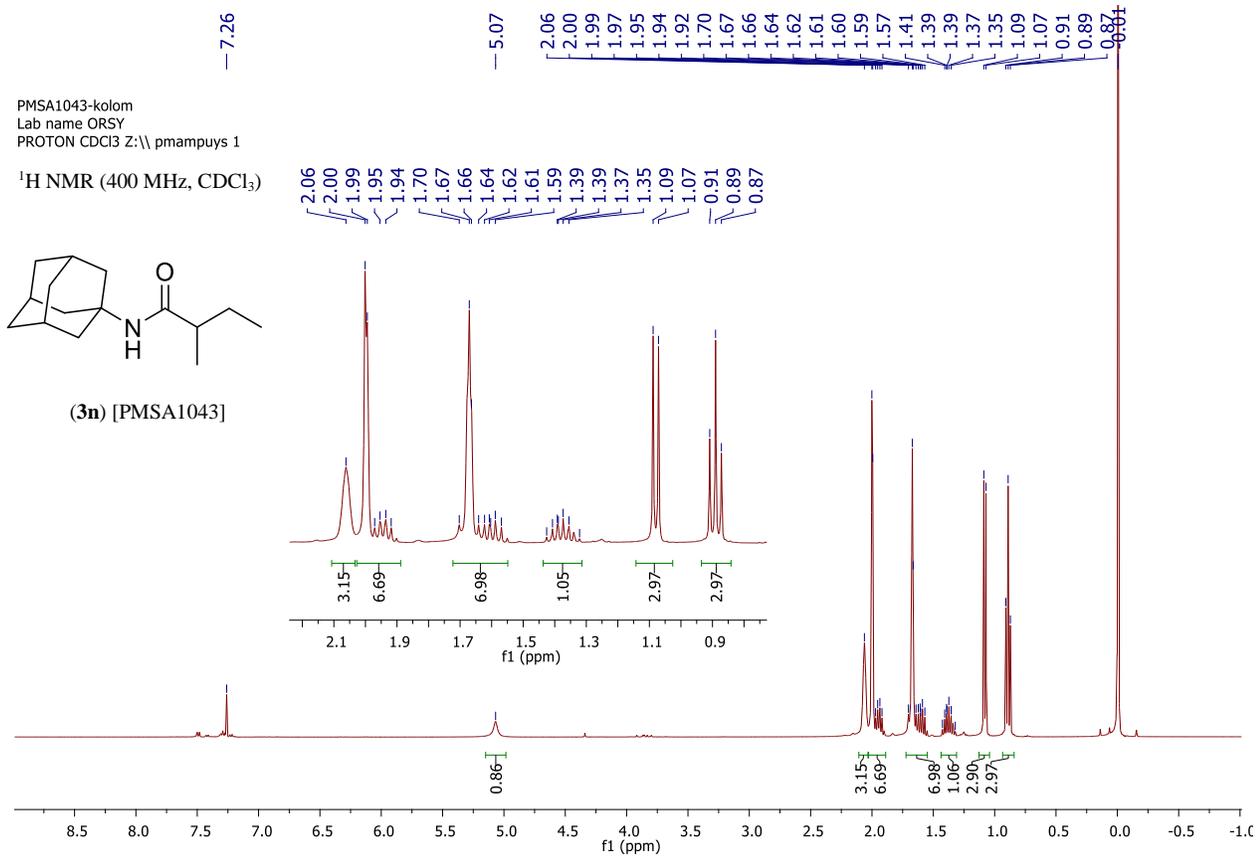
COSY NMR (400 MHz, CDCl₃)



PMSA949-APT
Lab name ORSY
COSY-Regular CDCl₃ Z:\\ pmampuys 7

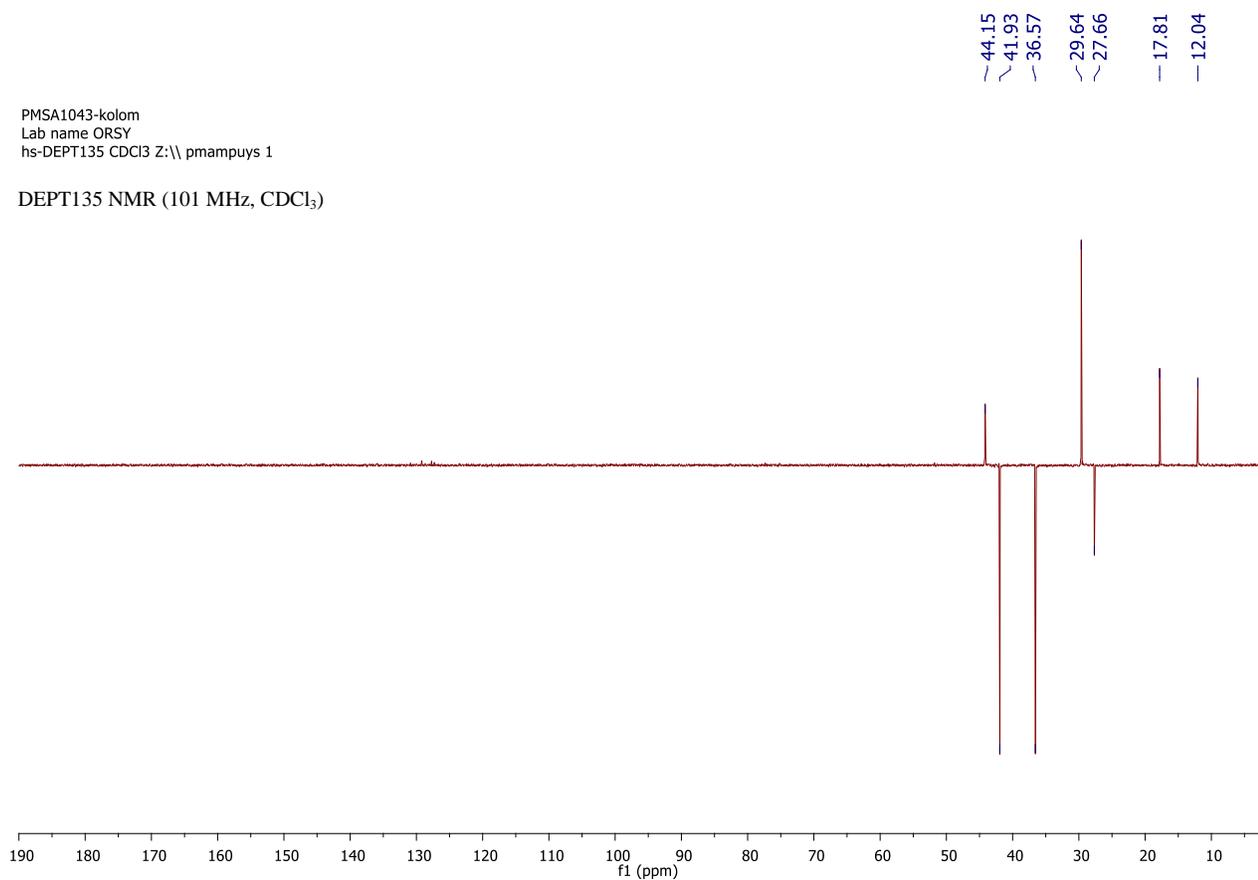


Copies of the NMR spectra

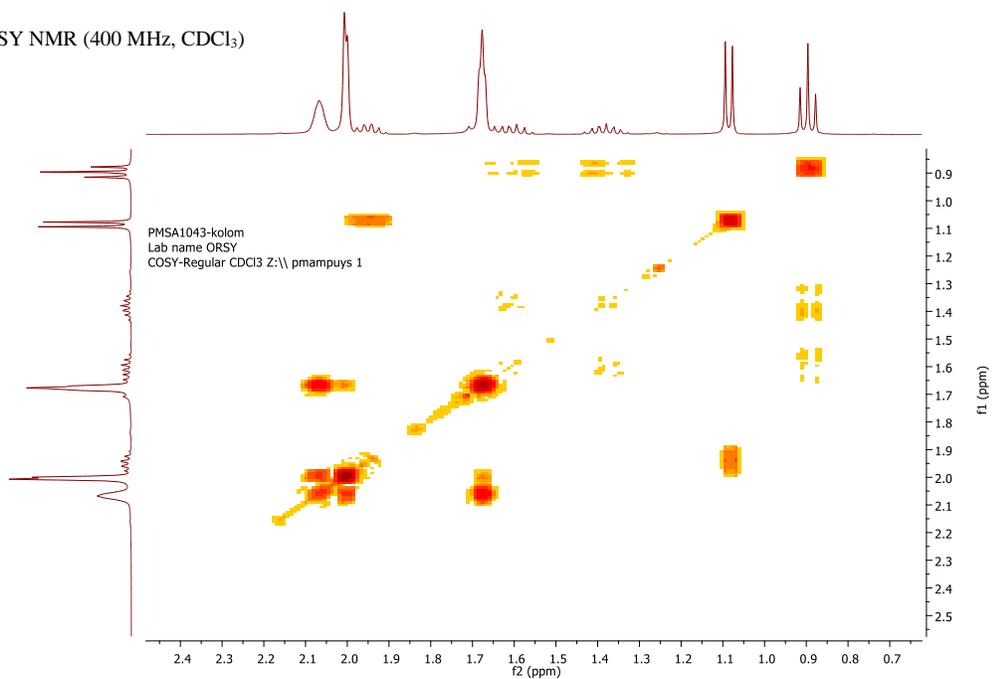


PMSA1043-kolom
 Lab name ORSY
 hs-DEPT135 CDCl3 Z:\\ pmampuy 1

DEPT135 NMR (101 MHz, CDCl₃)



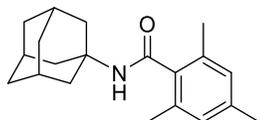
COSY NMR (400 MHz, CDCl₃)



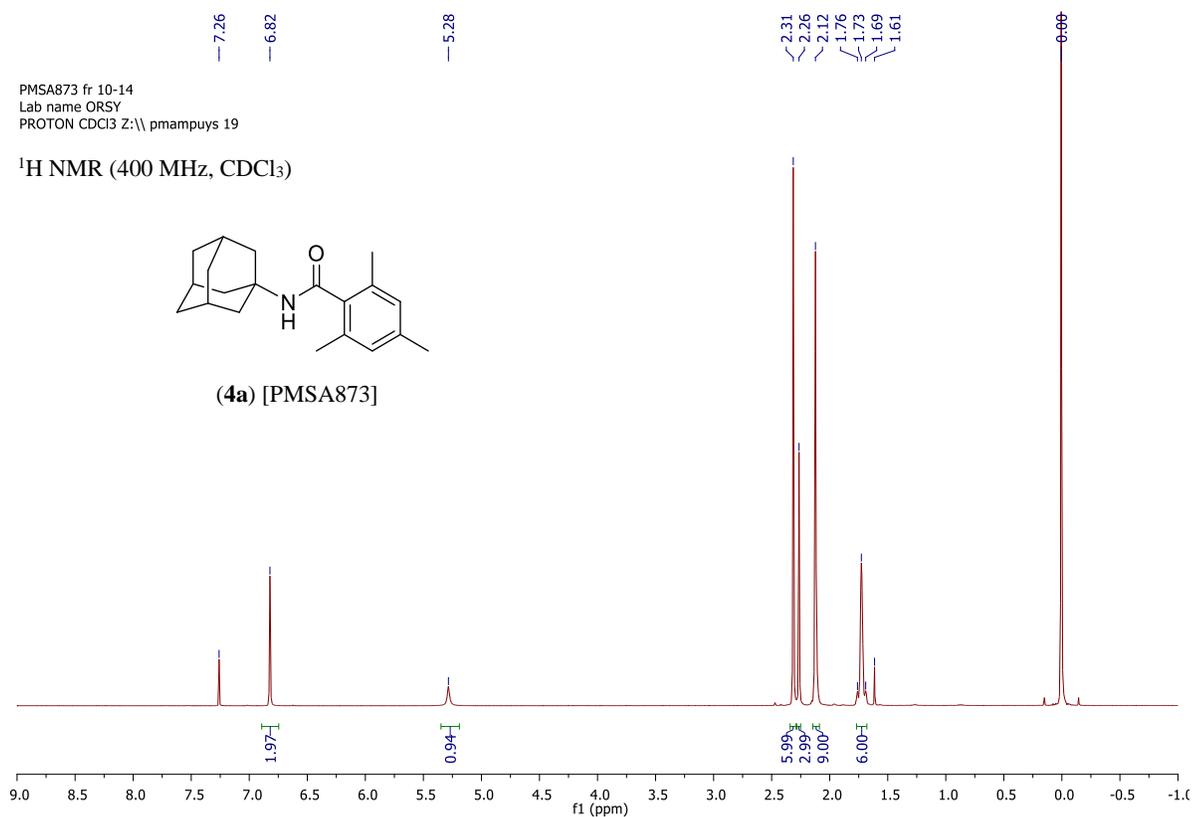
Copies of the NMR spectra

PMSA873 fr 10-14
 Lab name ORSY
 PROTON CDCl3 Z:\\ pmampuys 19

¹H NMR (400 MHz, CDCl₃)

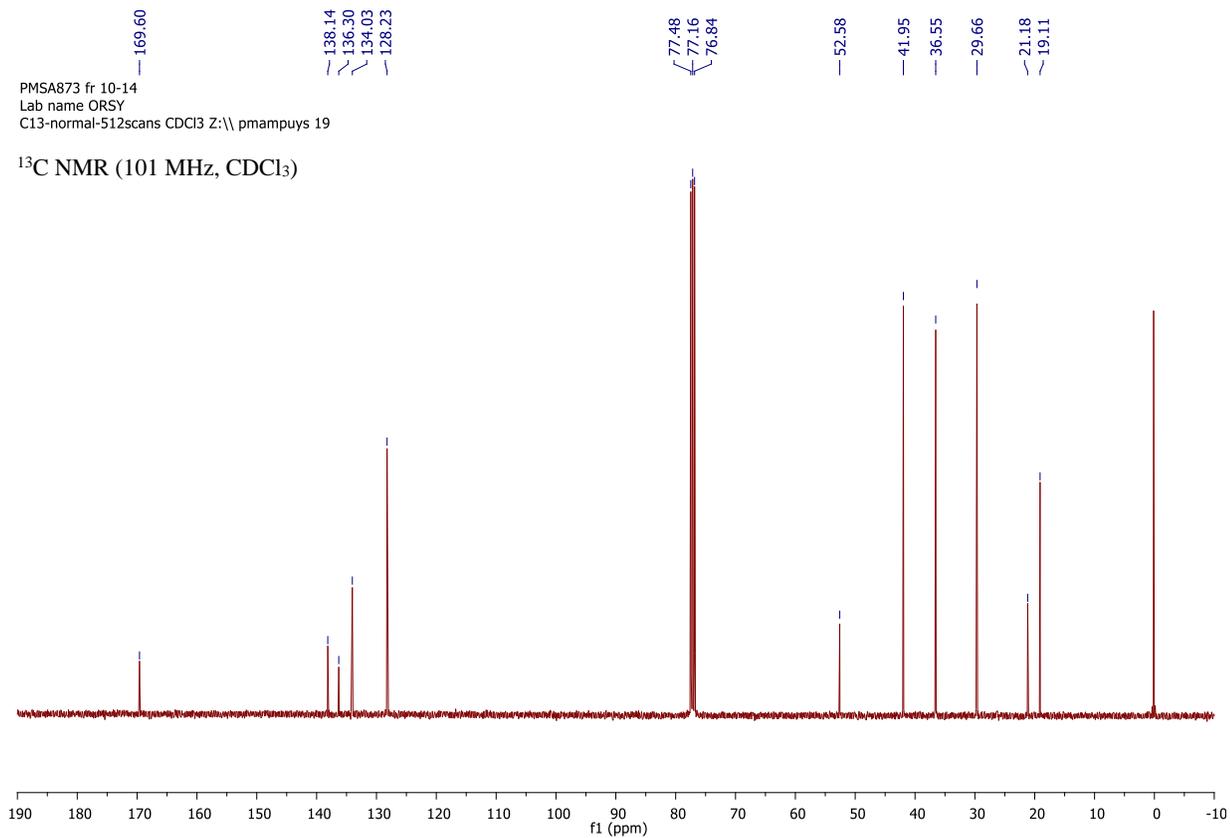


(4a) [PMSA873]



PMSA873 fr 10-14
 Lab name ORSY
 C13-normal-512scans CDCl3 Z:\\ pmampuys 19

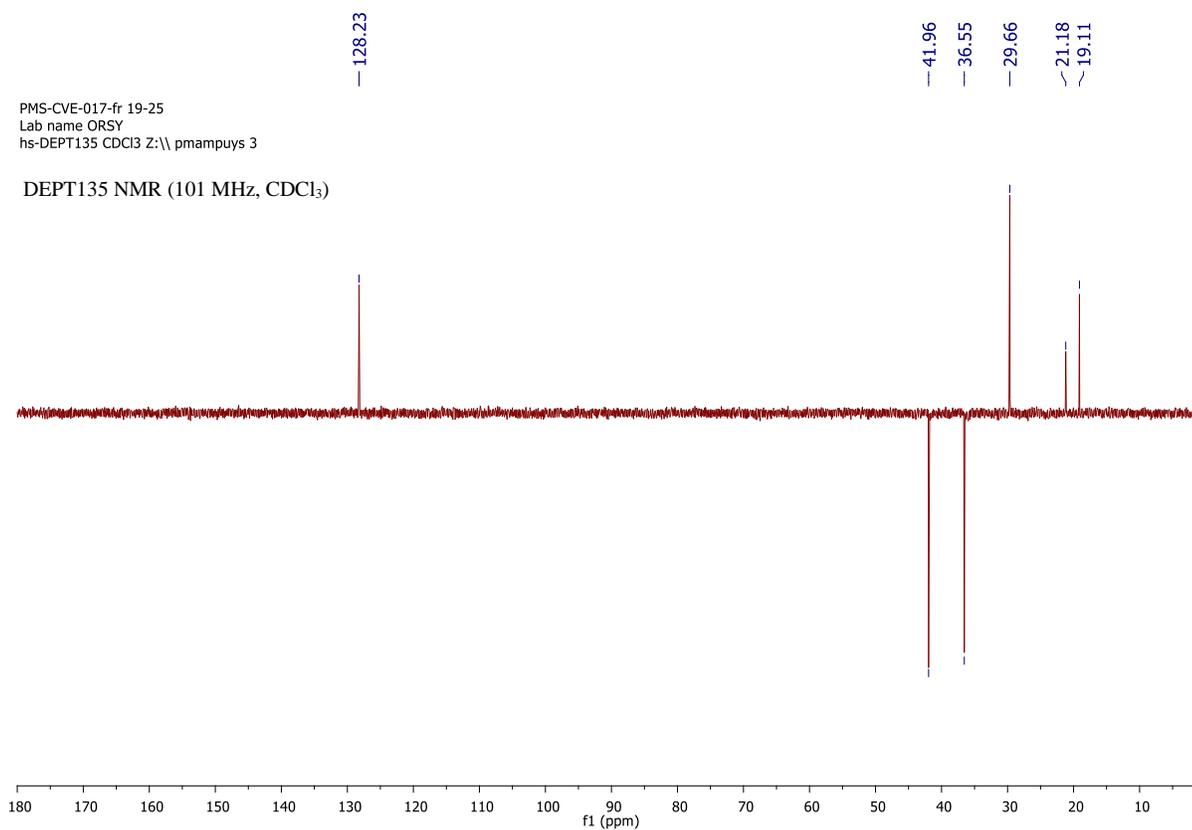
¹³C NMR (101 MHz, CDCl₃)



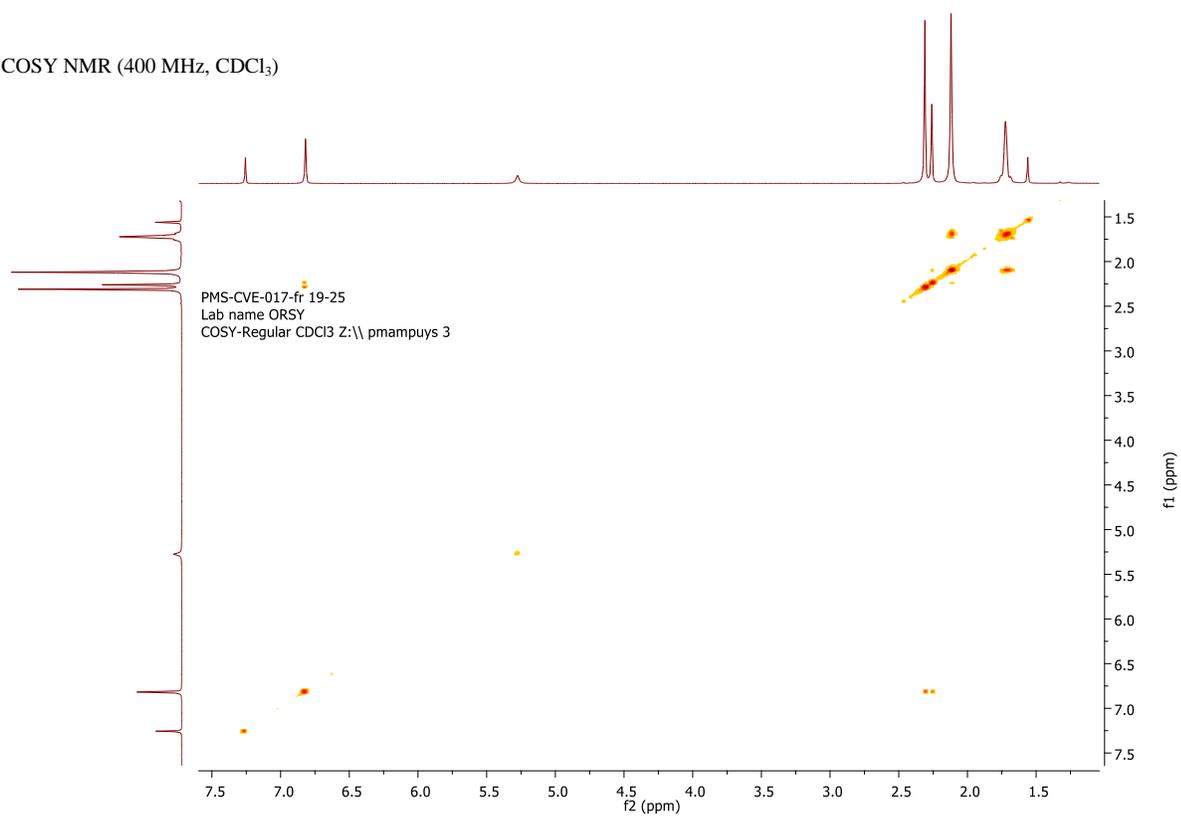
Copies of the NMR spectra

PMS-CVE-017-fr 19-25
Lab name ORSY
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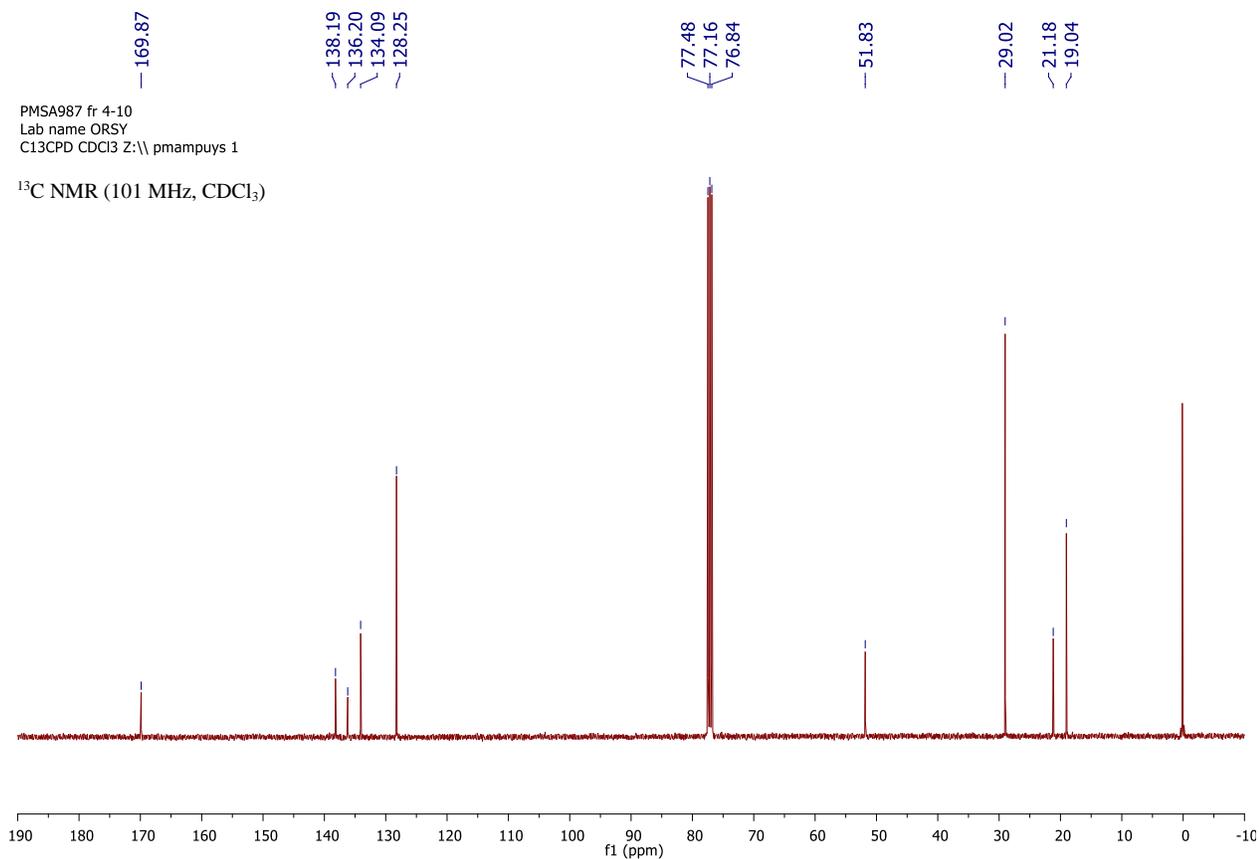
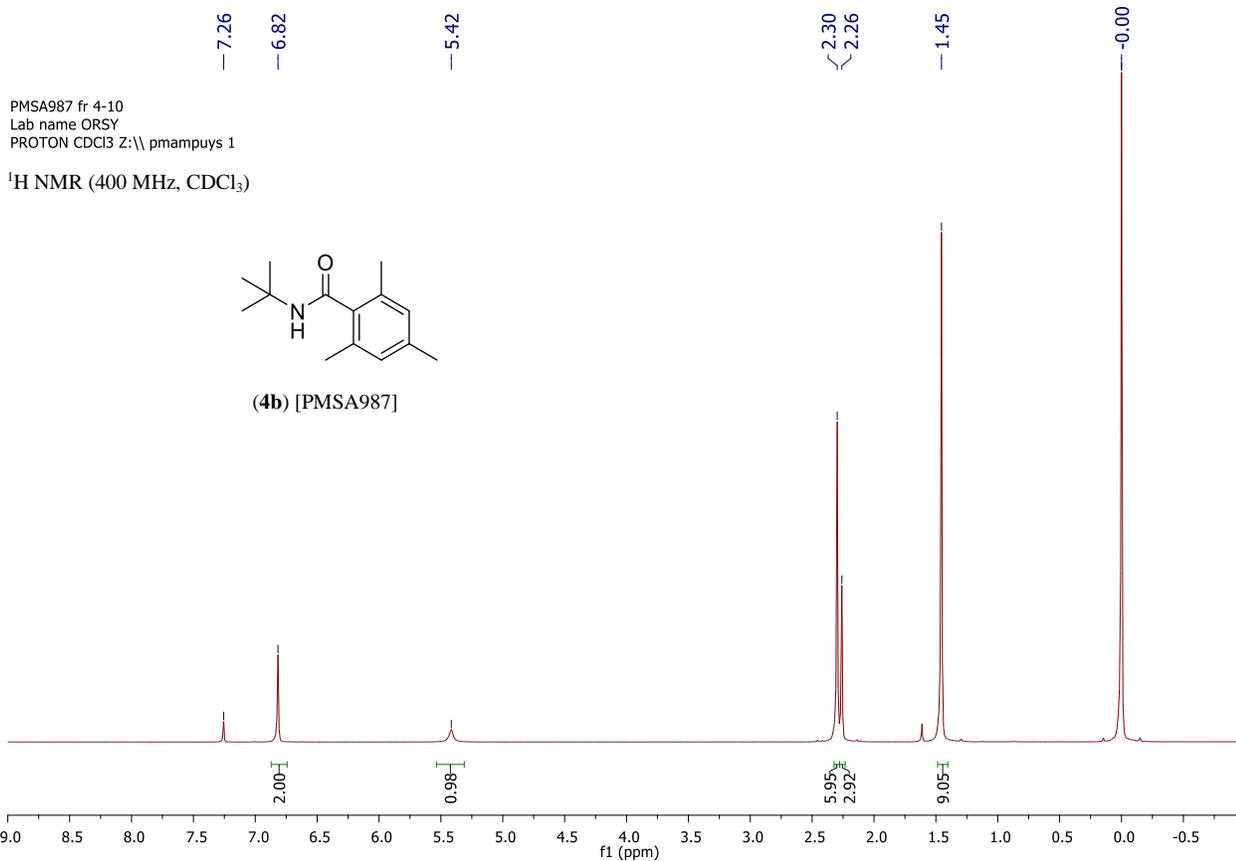
DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)



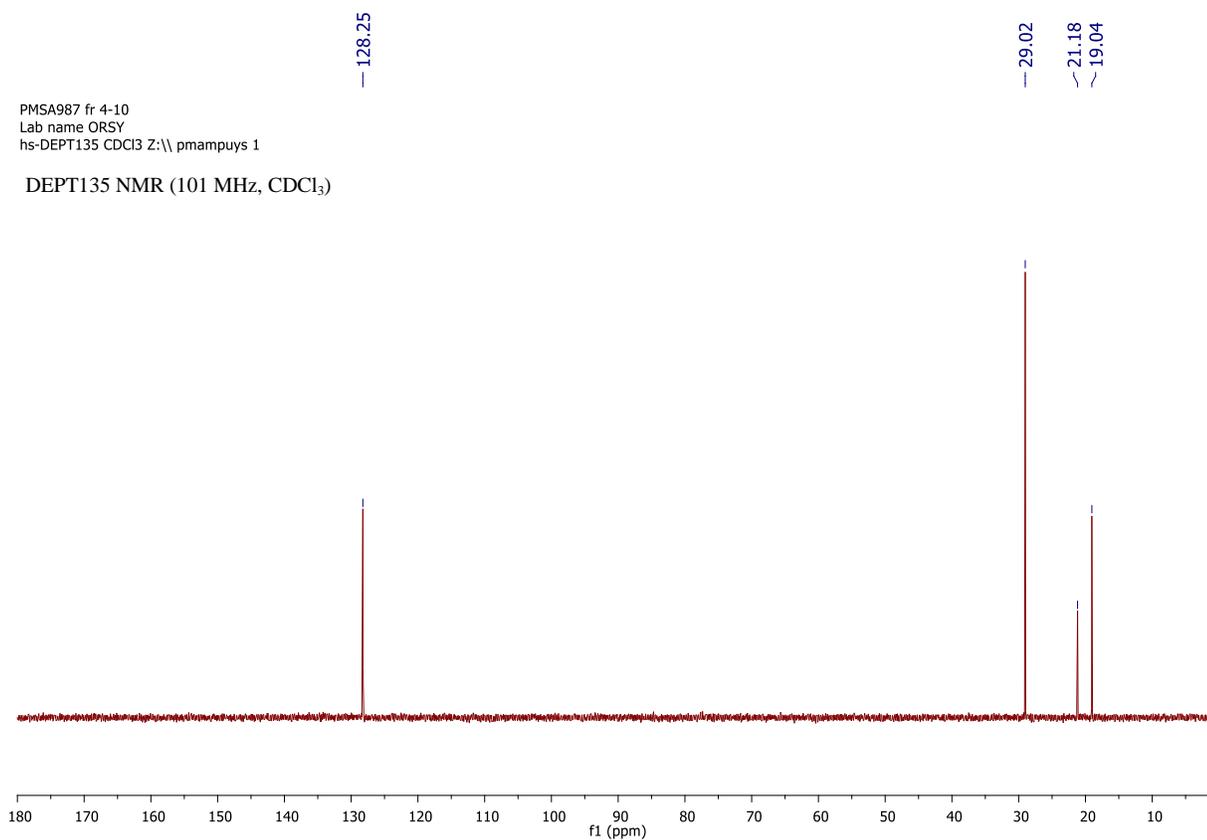
Copies of the NMR spectra



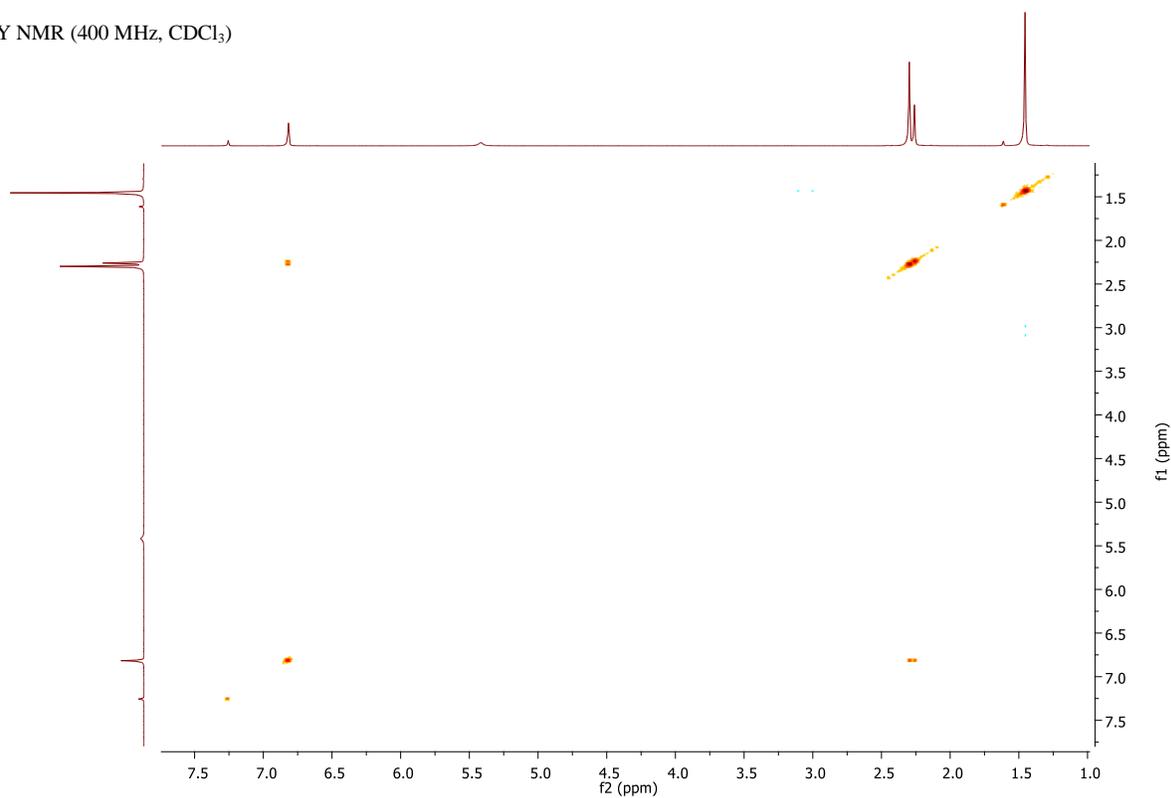
Copies of the NMR spectra

PMSA987 fr 4-10
Lab name ORSY
hs-DEPT135 CDCl3 Z:\\ pmampuys 1

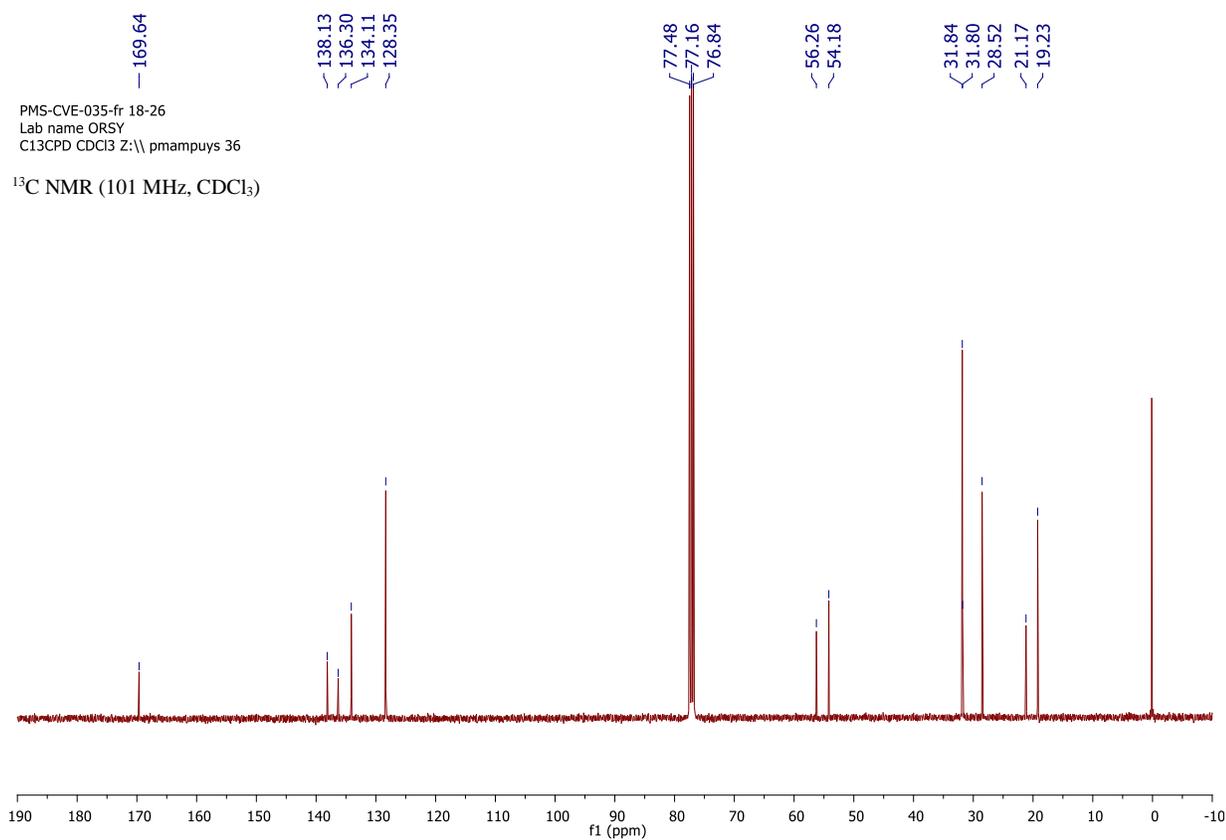
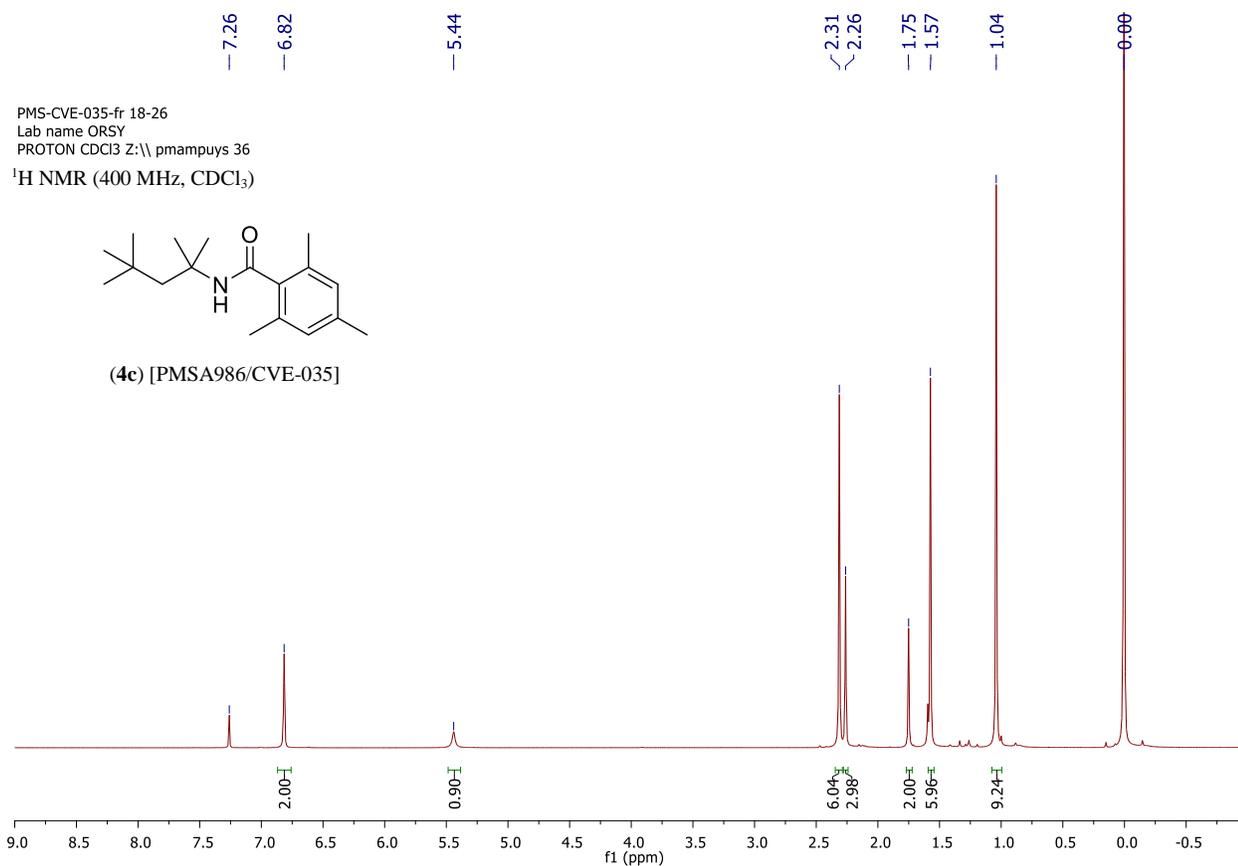
DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)



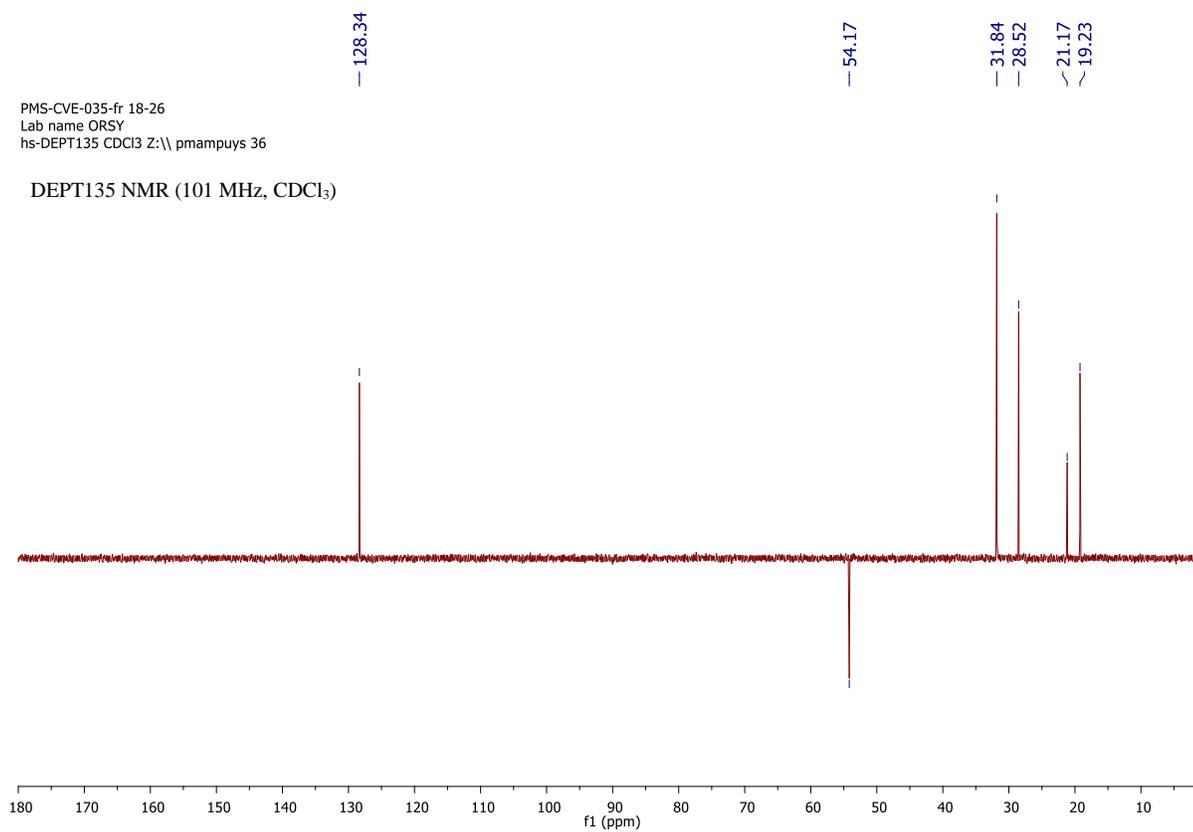
Copies of the NMR spectra



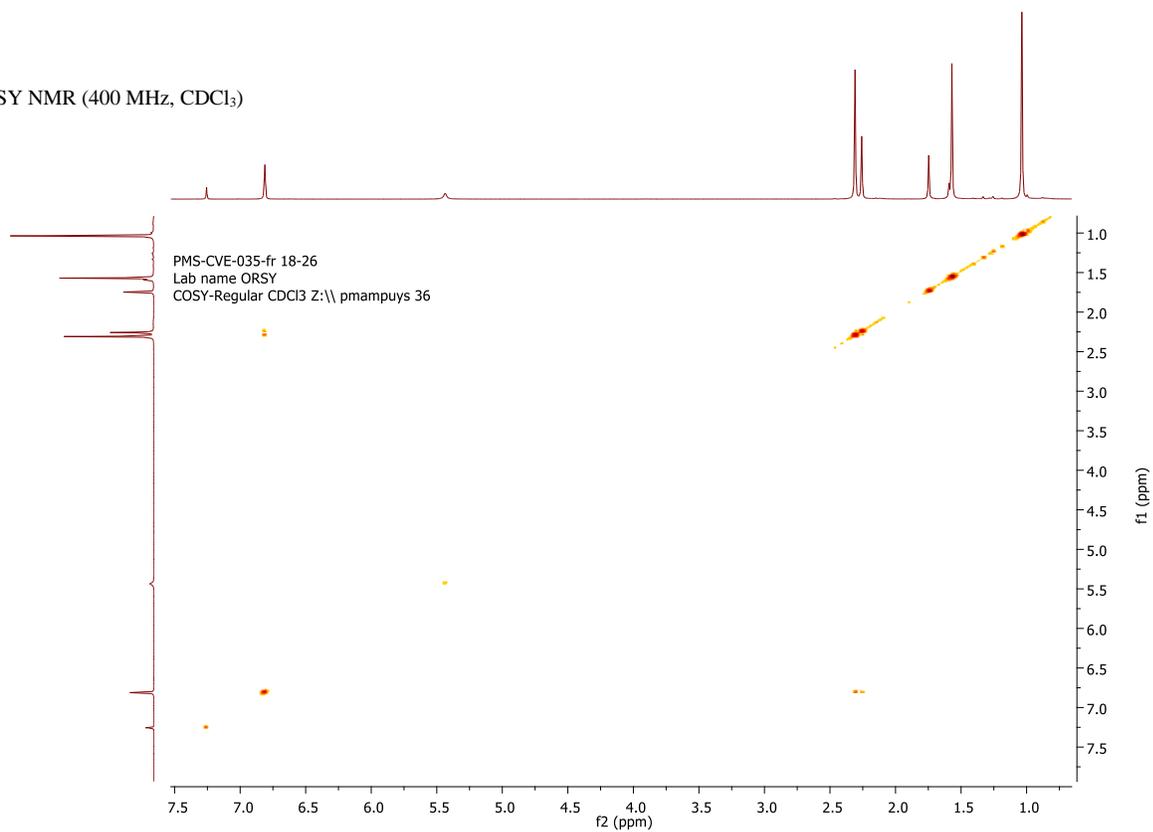
Copies of the NMR spectra

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Lab name ORSY
hs-DEPT135 CDCl3 Z:\\ pmampuys 36

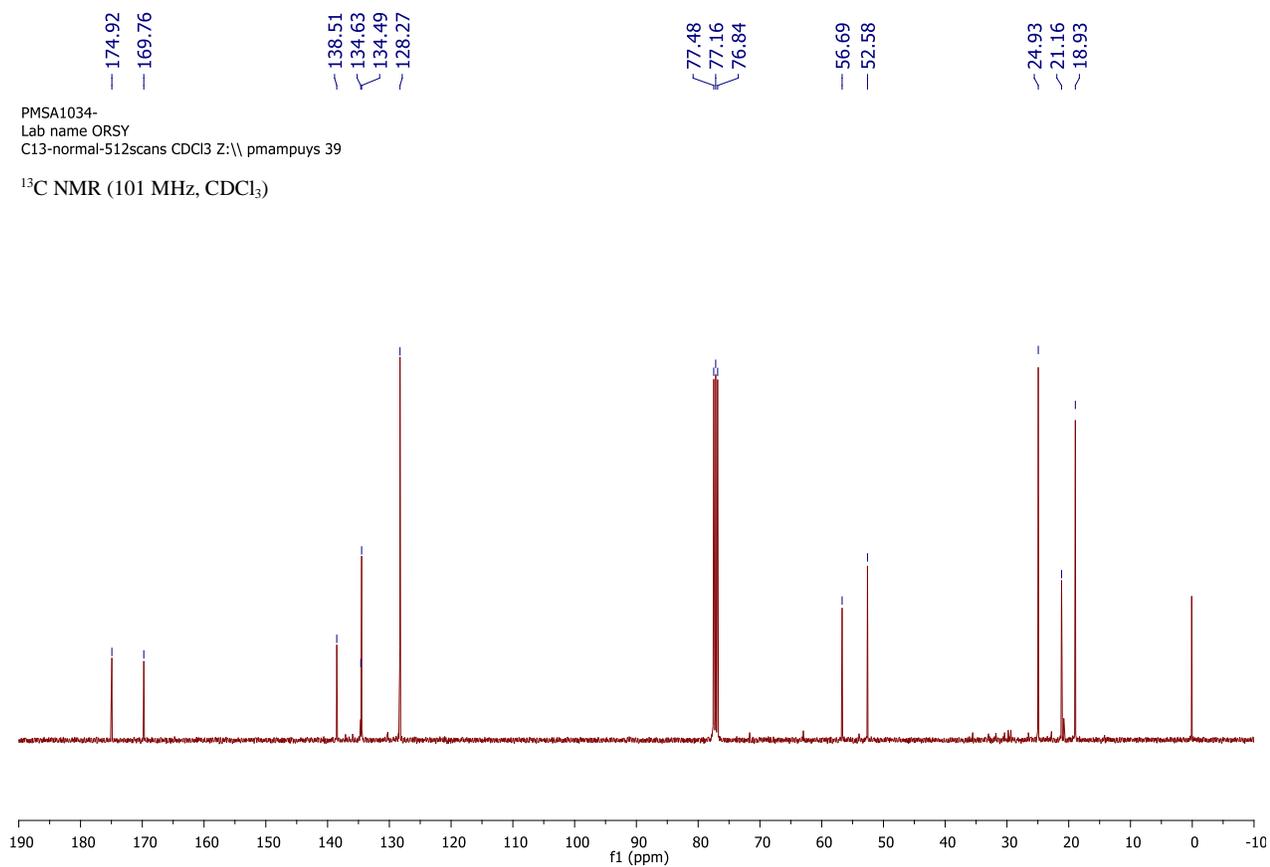
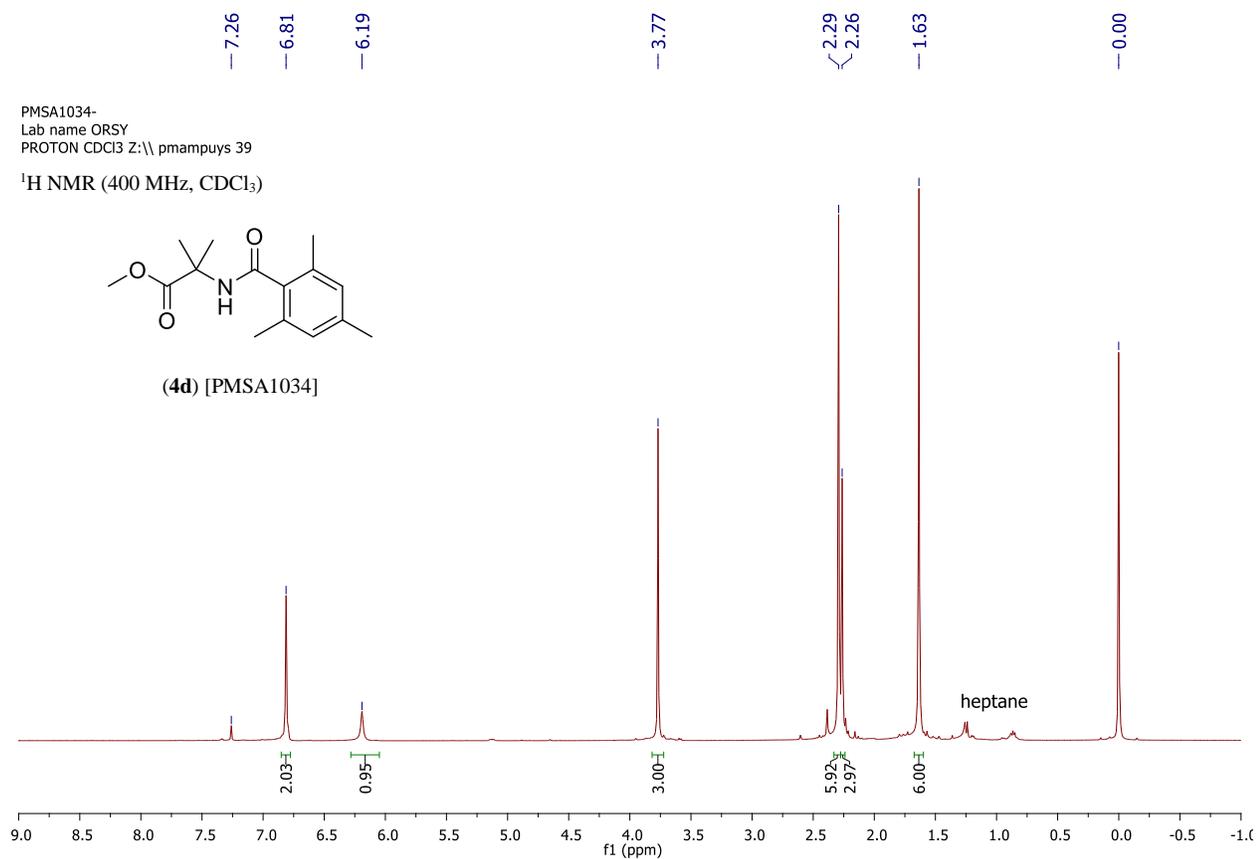
DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)

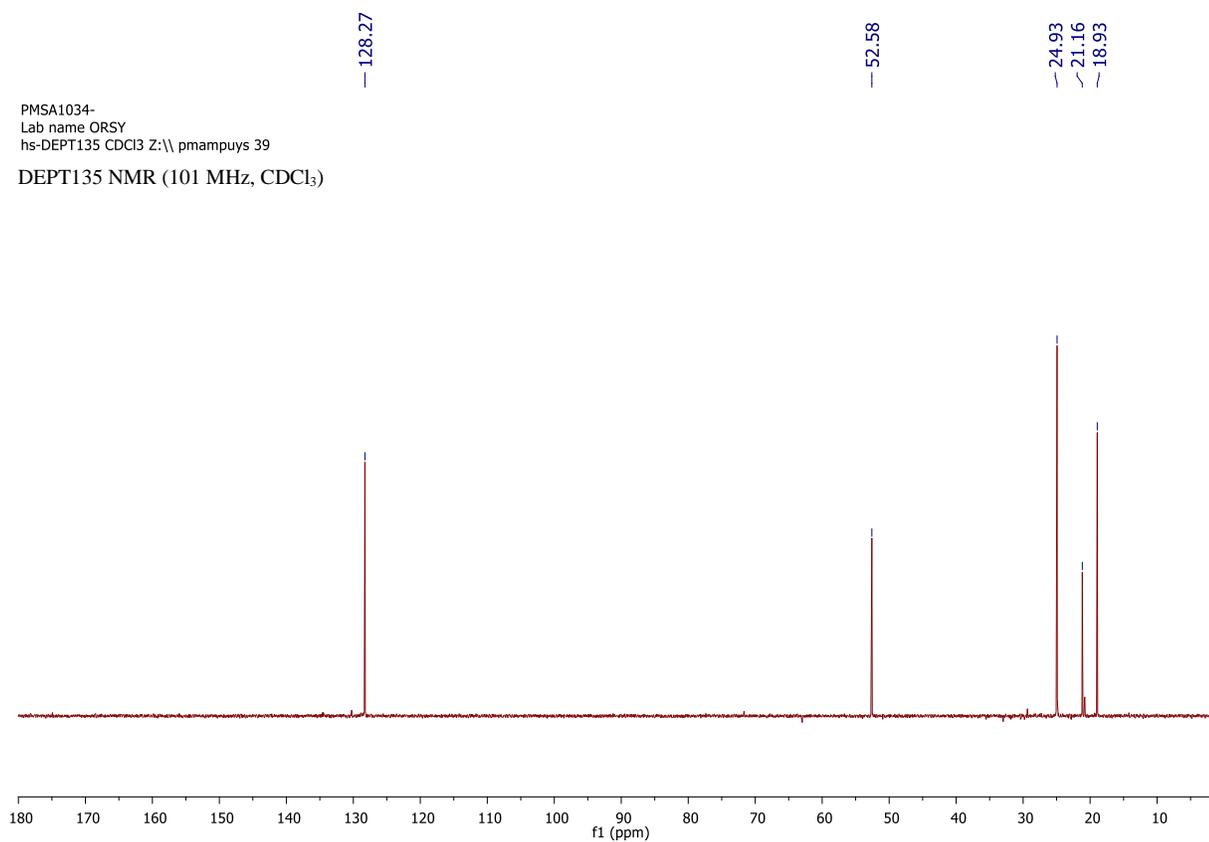


Copies of the NMR spectra

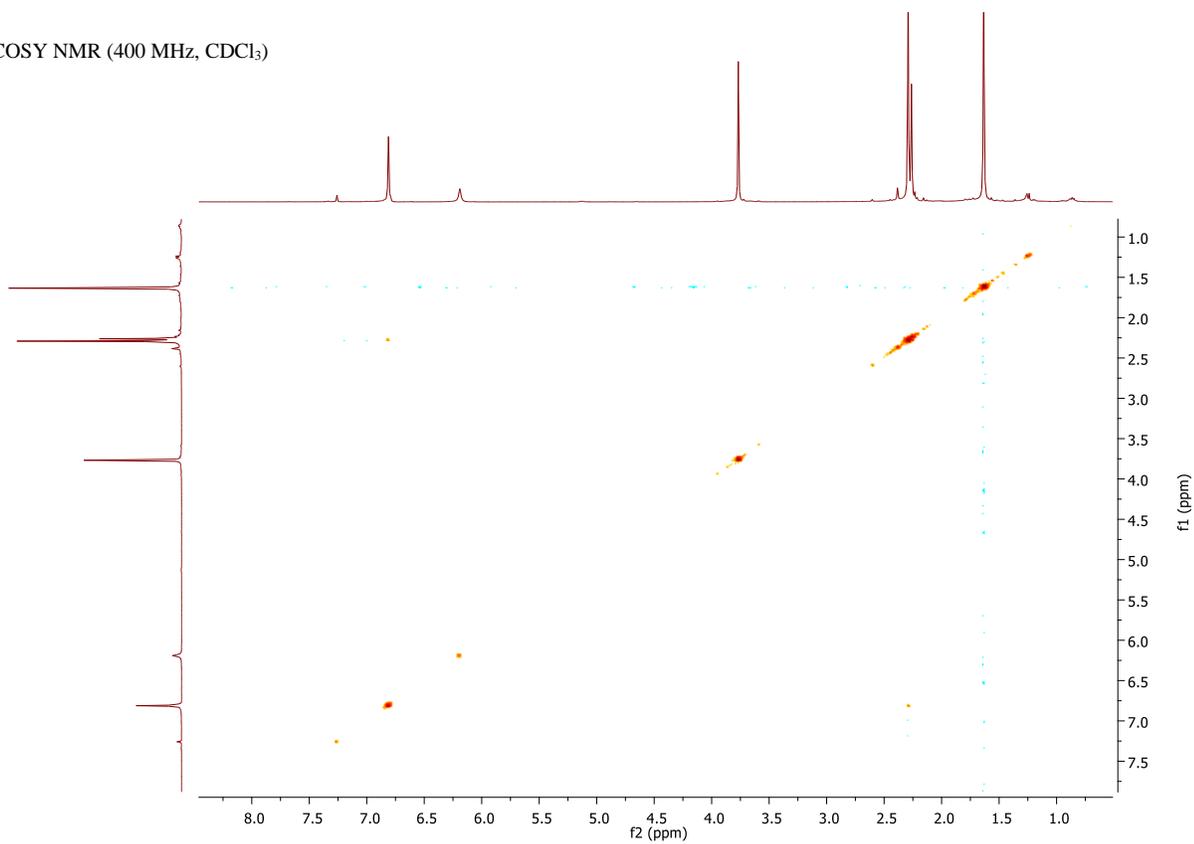


Copies of the NMR spectra

PMSA1034-
Lab name ORSY
hs-DEPT135 CDCl3 Z:\\ pmampuys 39
DEPT135 NMR (101 MHz, CDCl₃)



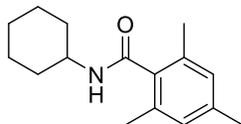
COSY NMR (400 MHz, CDCl₃)



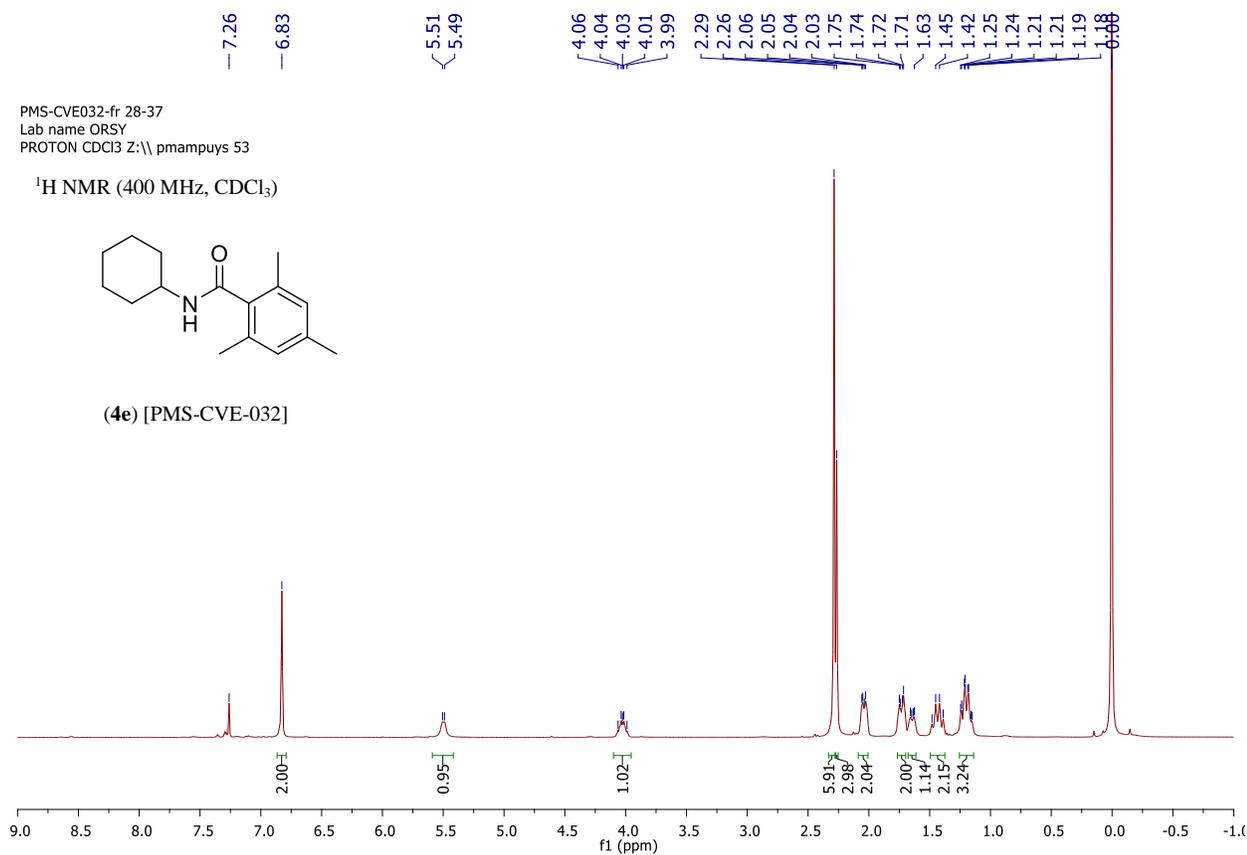
Copies of the NMR spectra

PMS-CVE032-fr 28-37
 Lab name ORSY
 PROTON CDCl3 Z:\ pmampuy5 53

¹H NMR (400 MHz, CDCl₃)

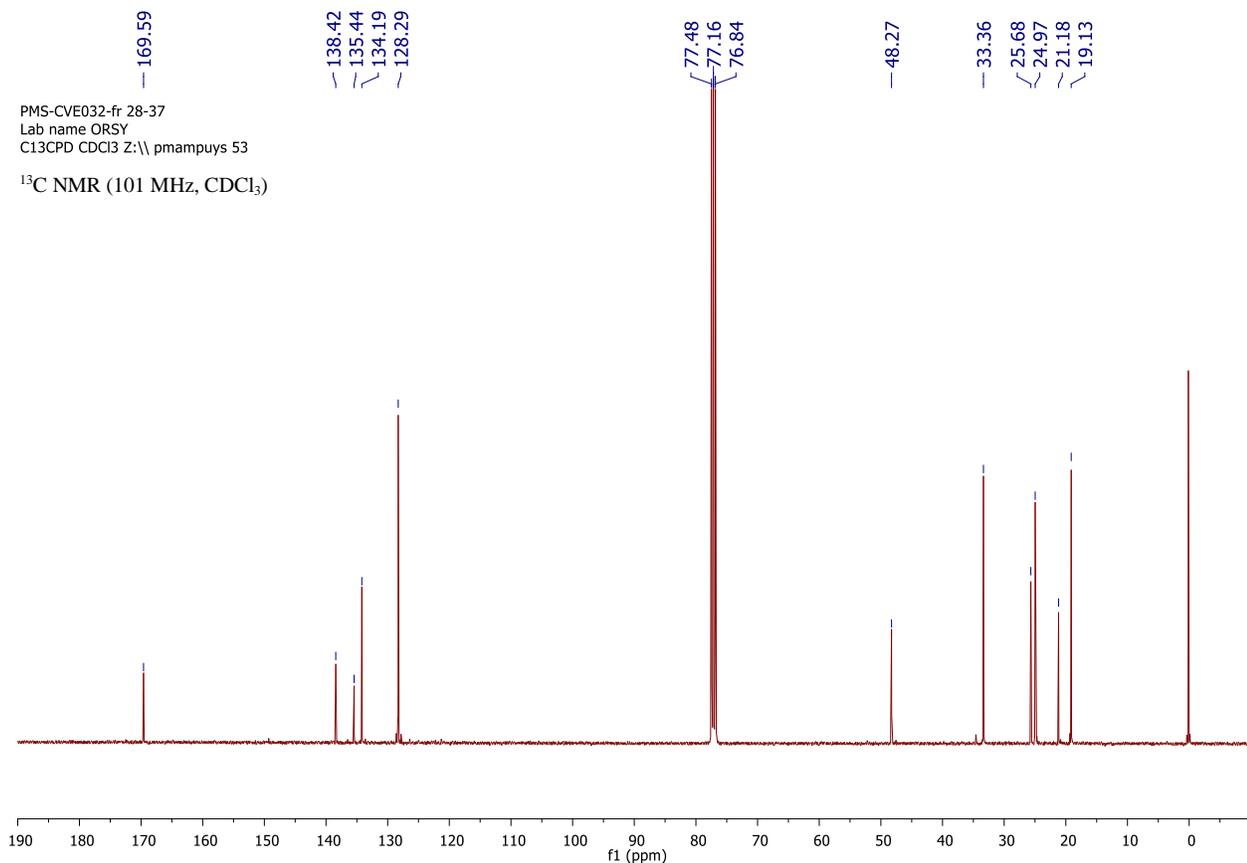


(4e) [PMS-CVE-032]

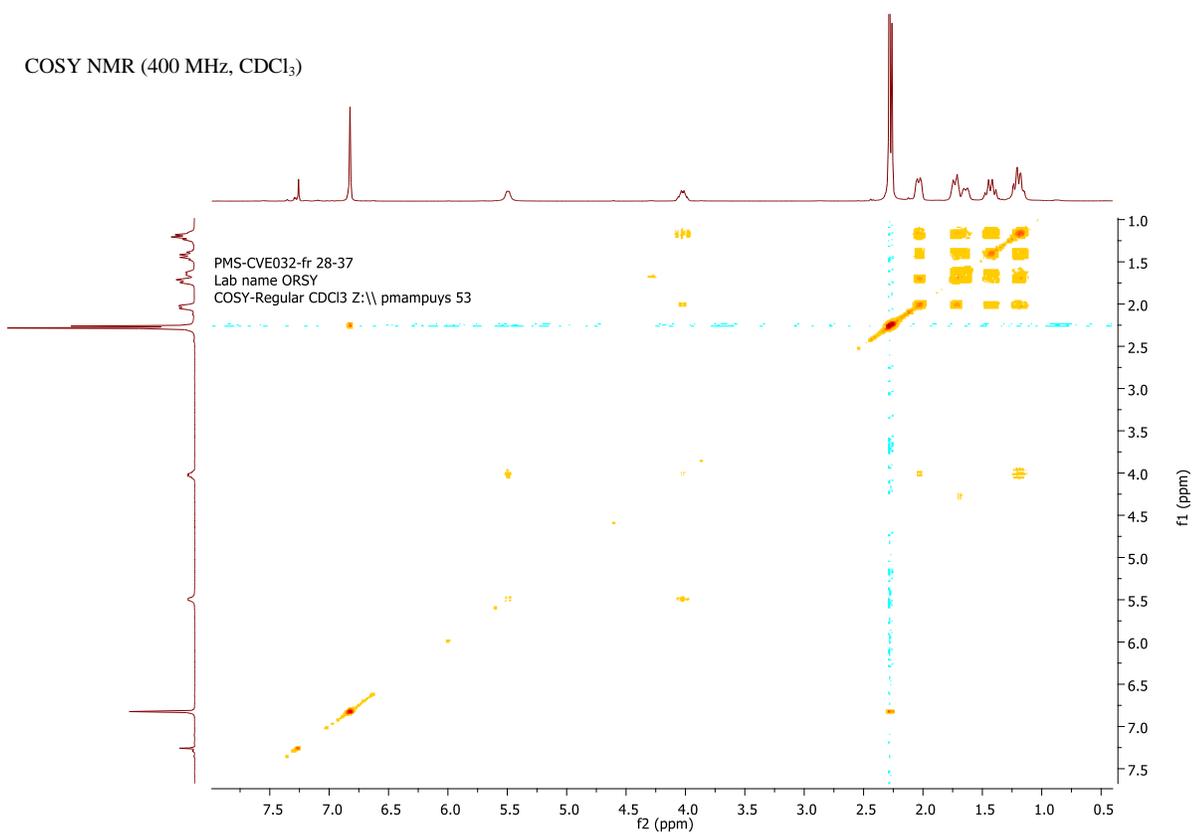
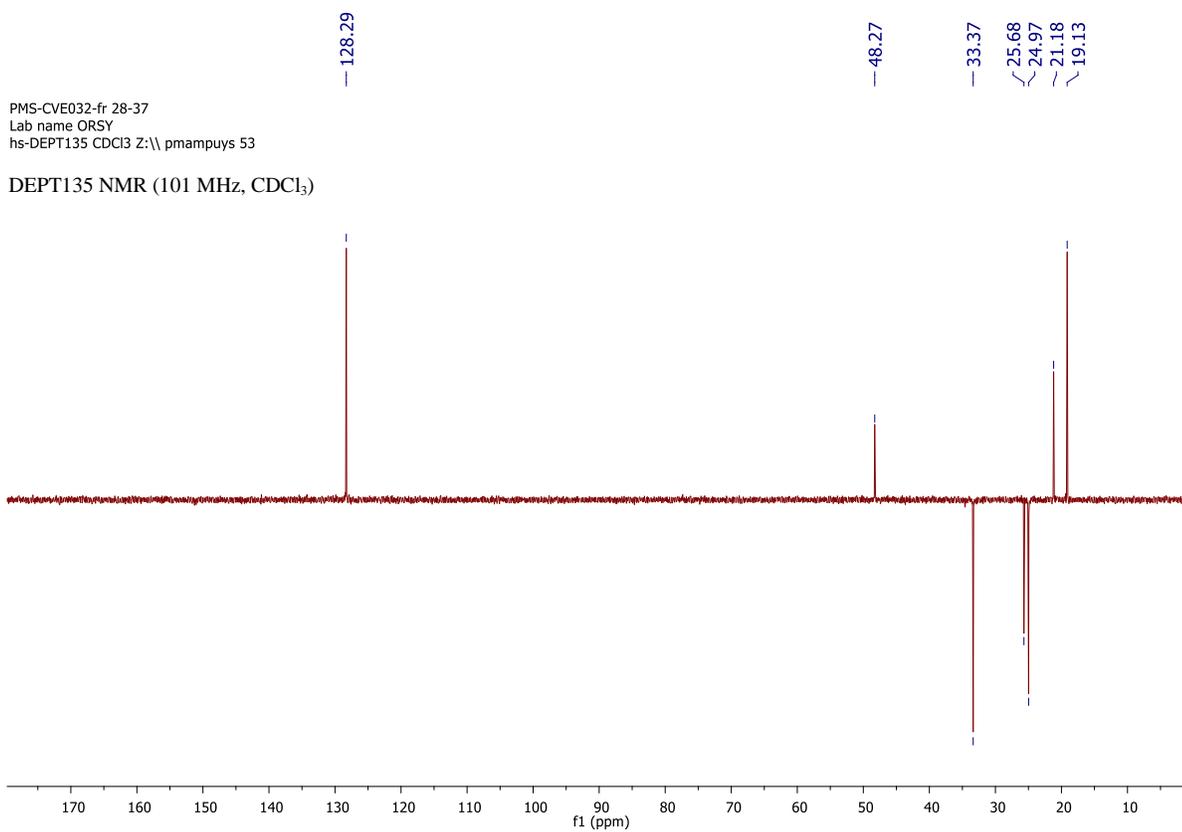


PMS-CVE032-fr 28-37
 Lab name ORSY
 C13CPD CDCl3 Z:\ pmampuy5 53

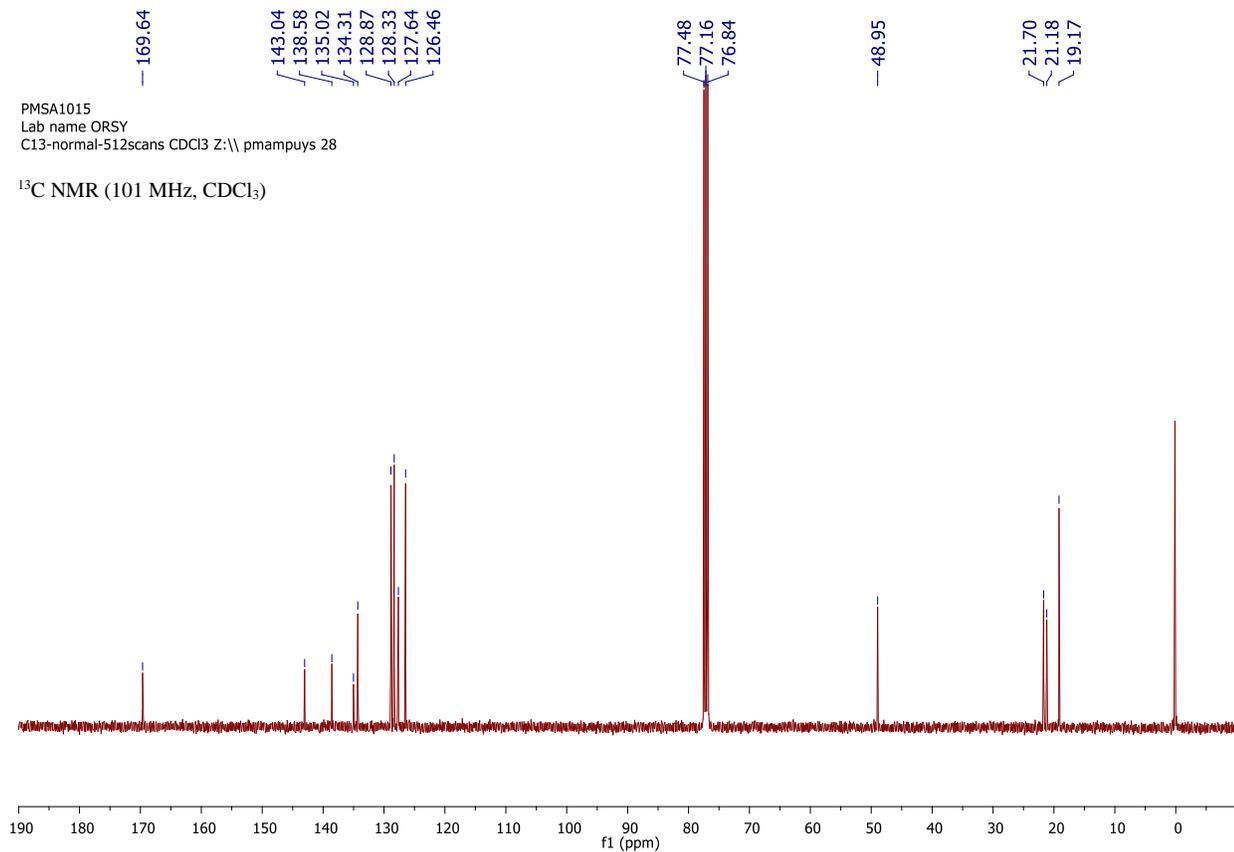
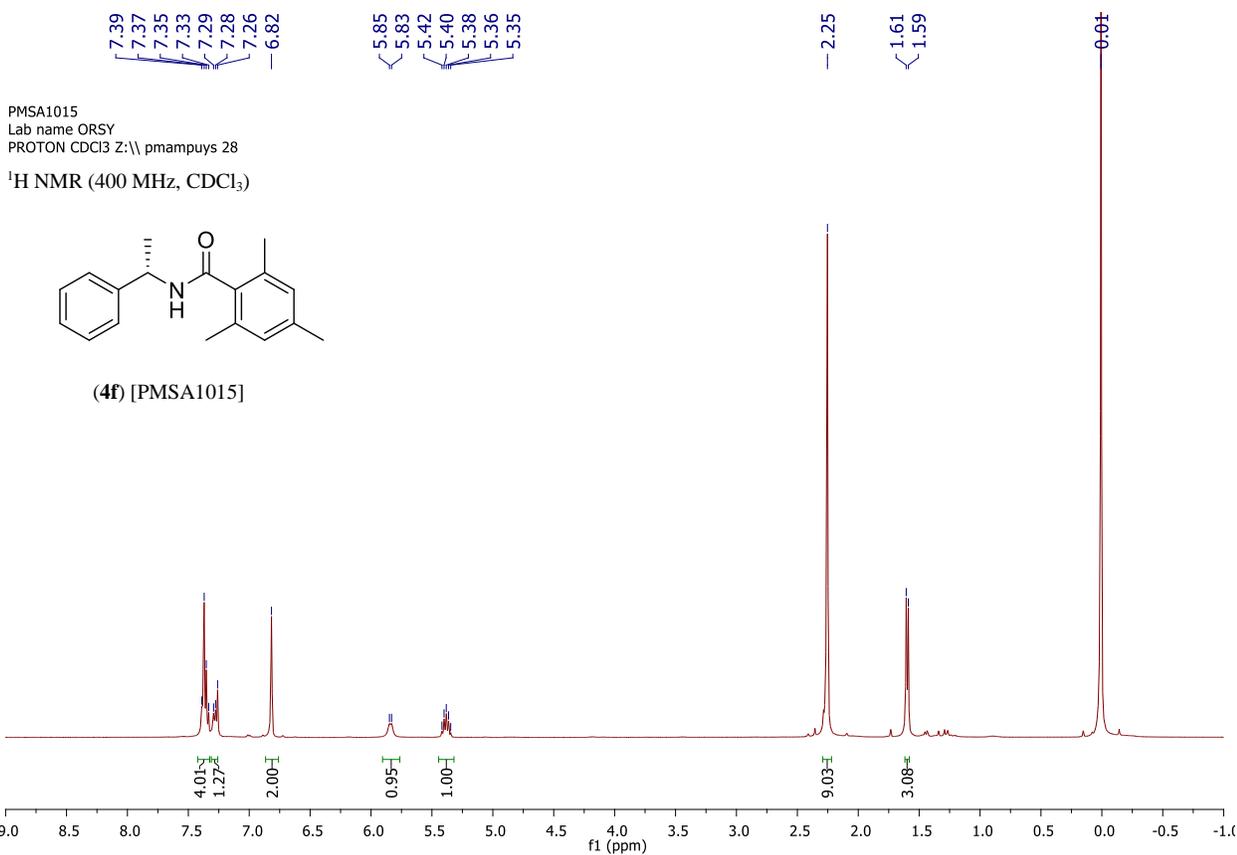
¹³C NMR (101 MHz, CDCl₃)



Copies of the NMR spectra



Copies of the NMR spectra



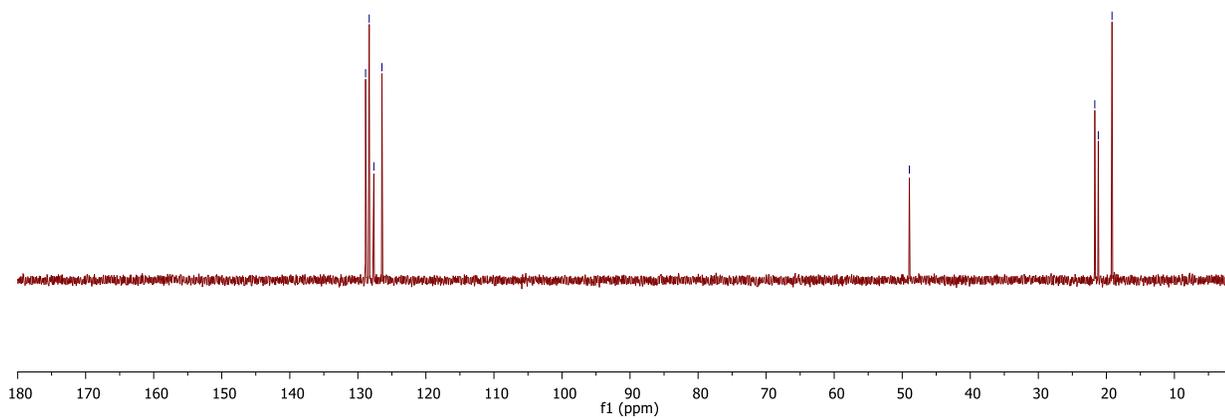
PMSA1015
Lab name ORSY
hs-DEPT135 CDCl3 Z:\\ pmampuys 28

DEPT135 NMR (101 MHz, CDCl₃)

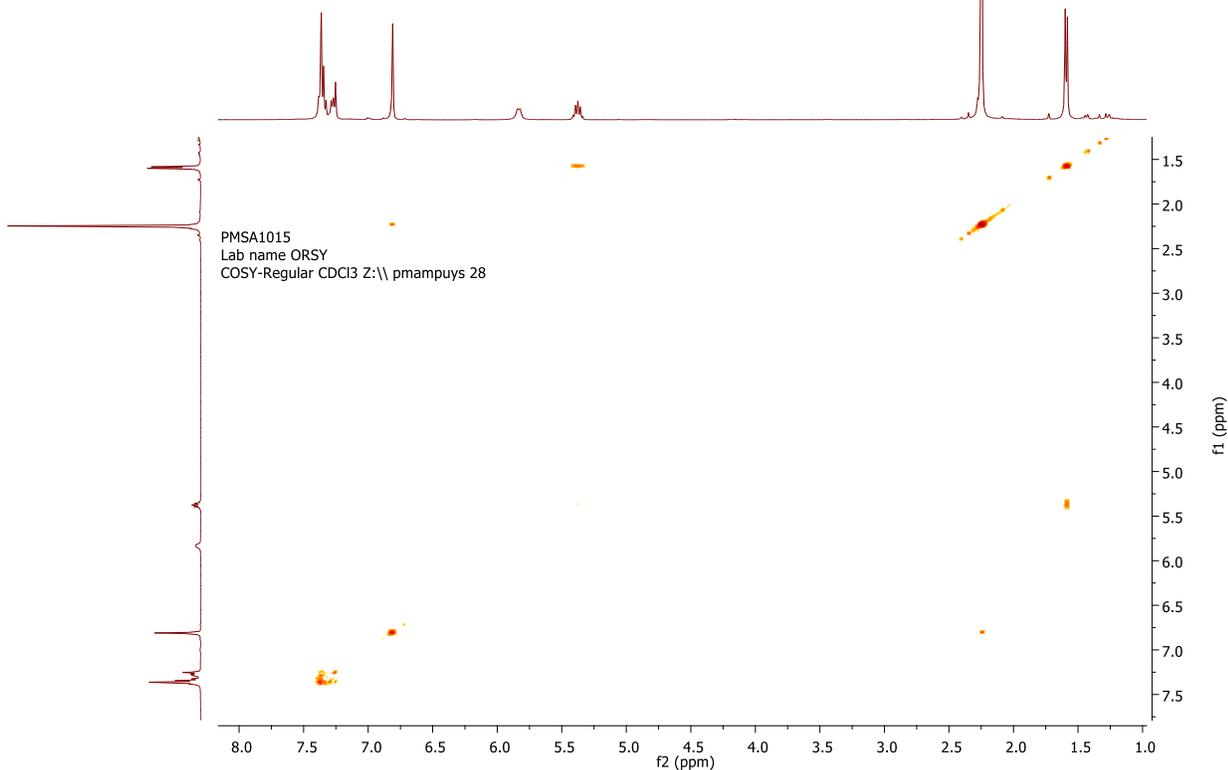
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48.95

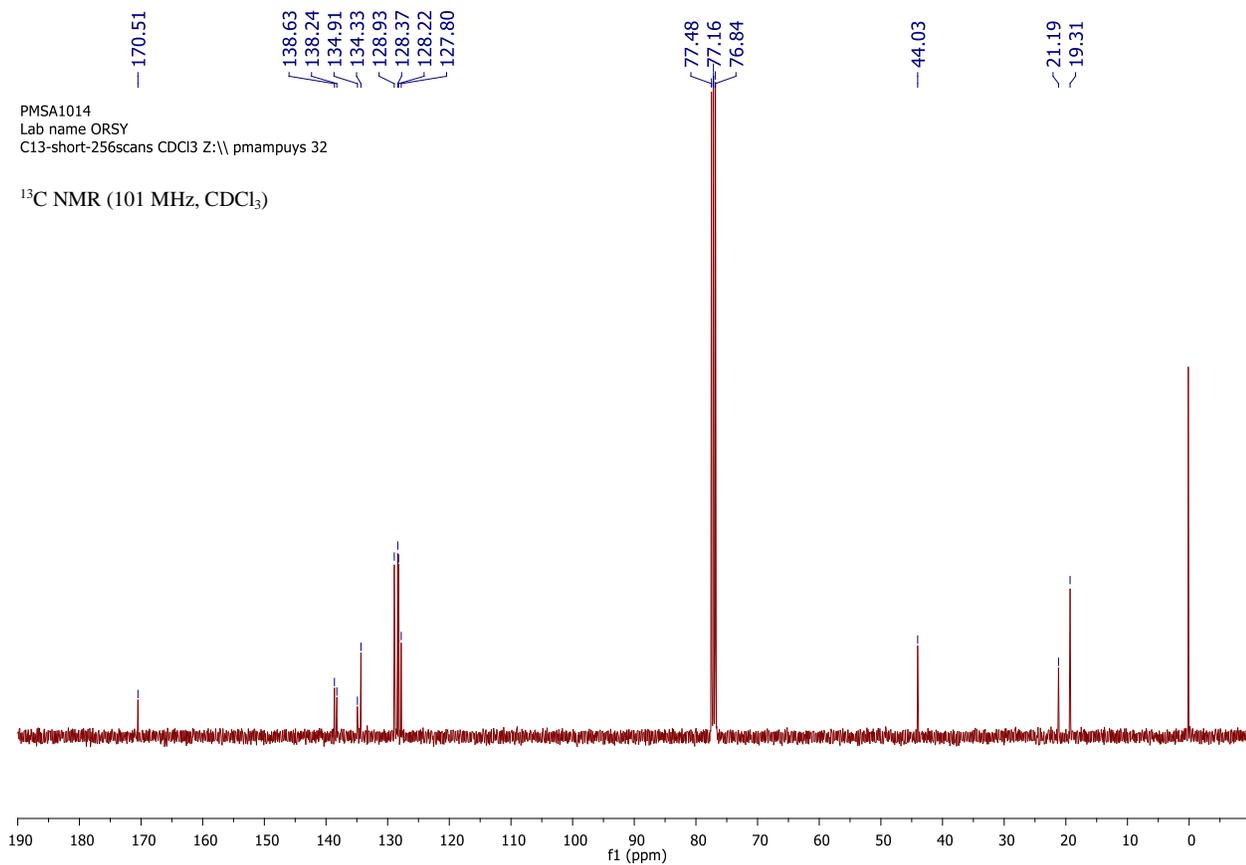
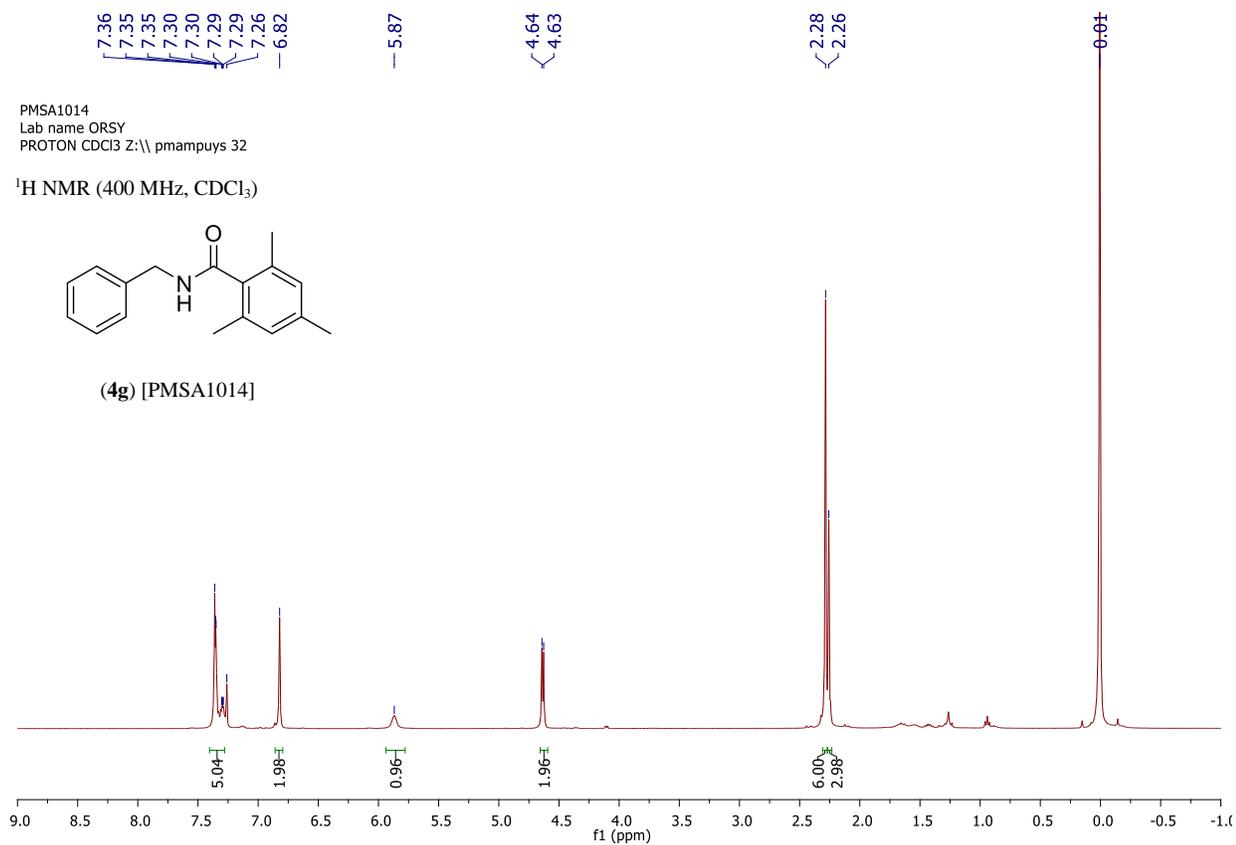
21.70
21.18
19.17



COSY NMR (400 MHz, CDCl₃)



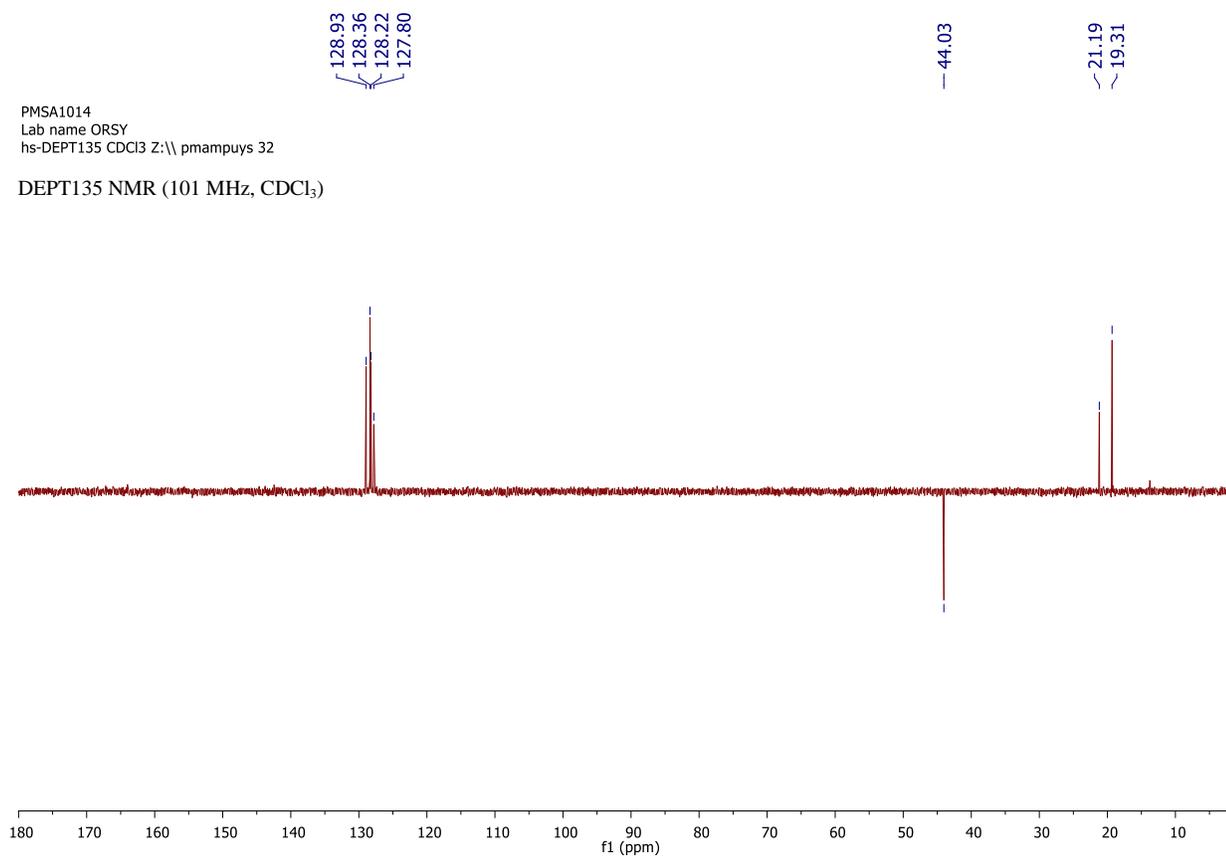
Copies of the NMR spectra



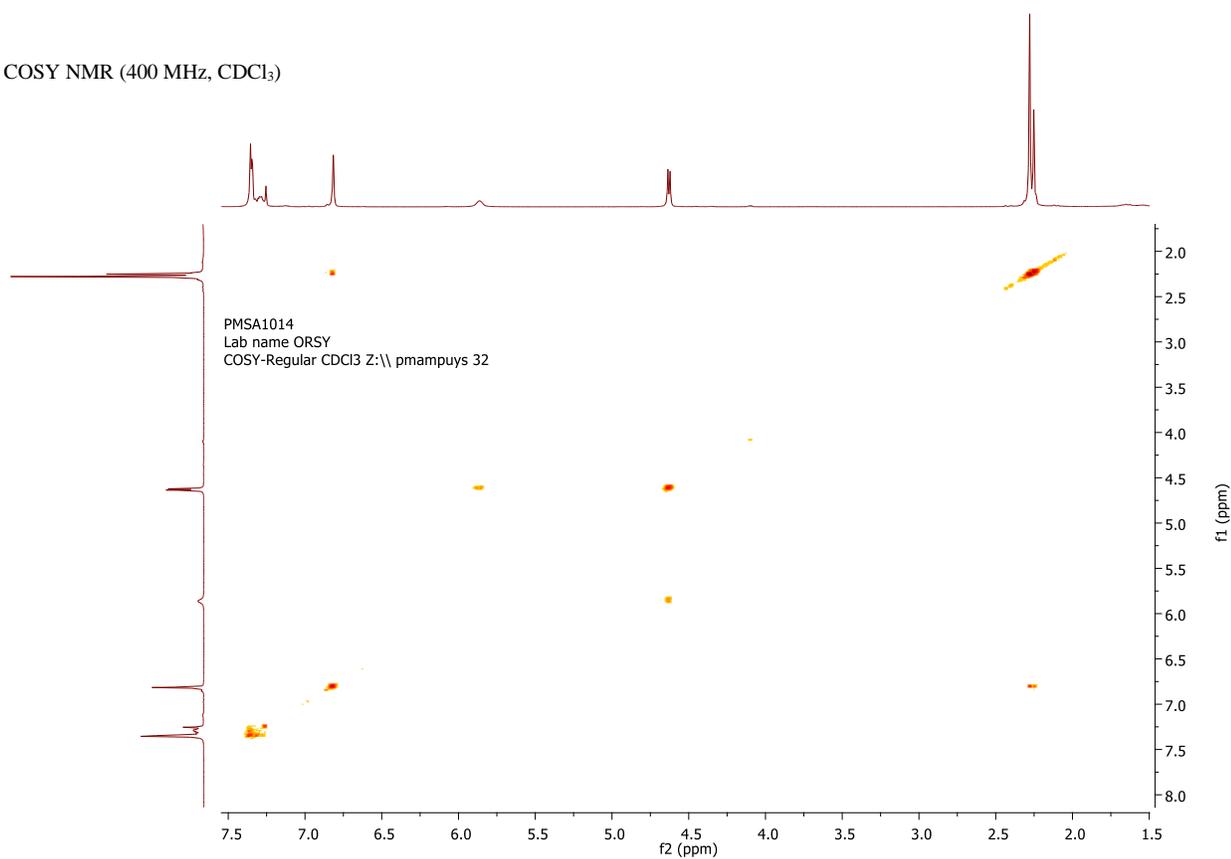
Copies of the NMR spectra

PMSA1014
Lab name ORSY
hs-DEPT135 CDCl₃ Z:\\ pmampuys 32

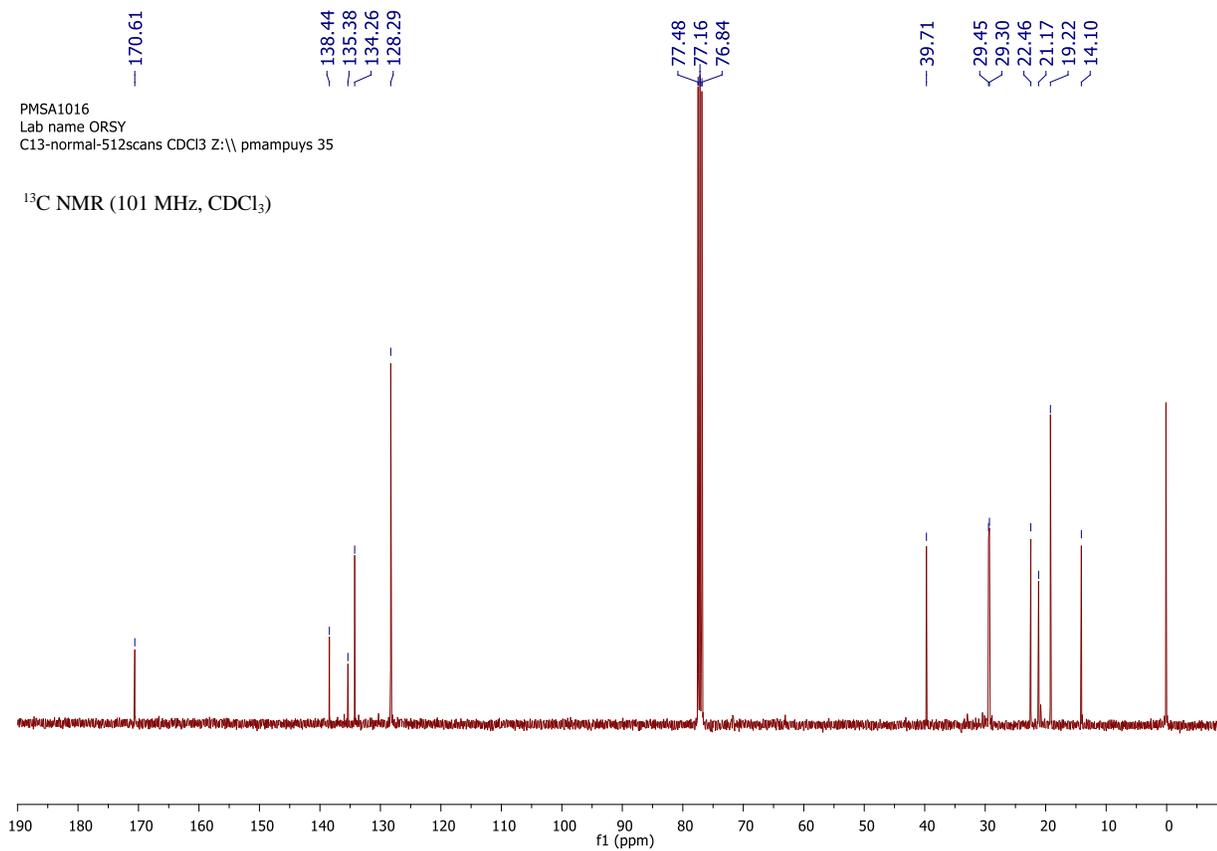
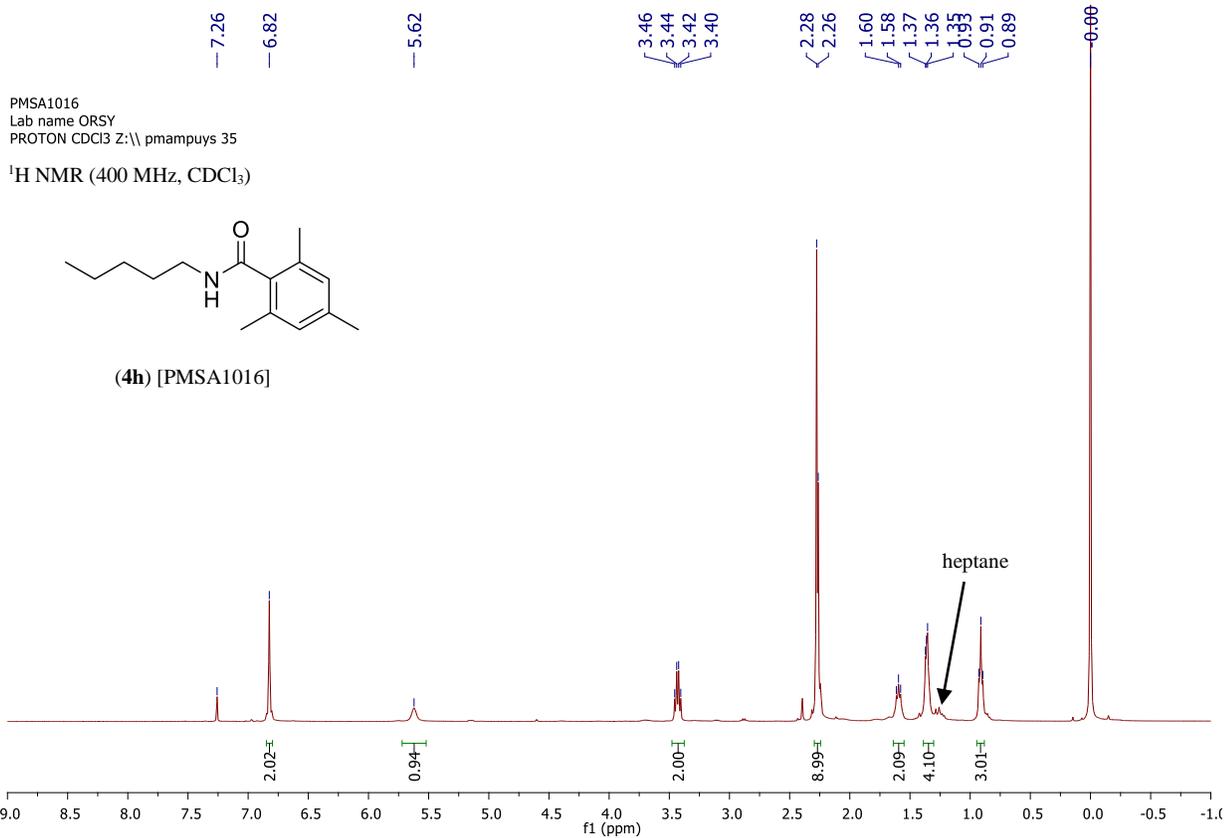
DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)



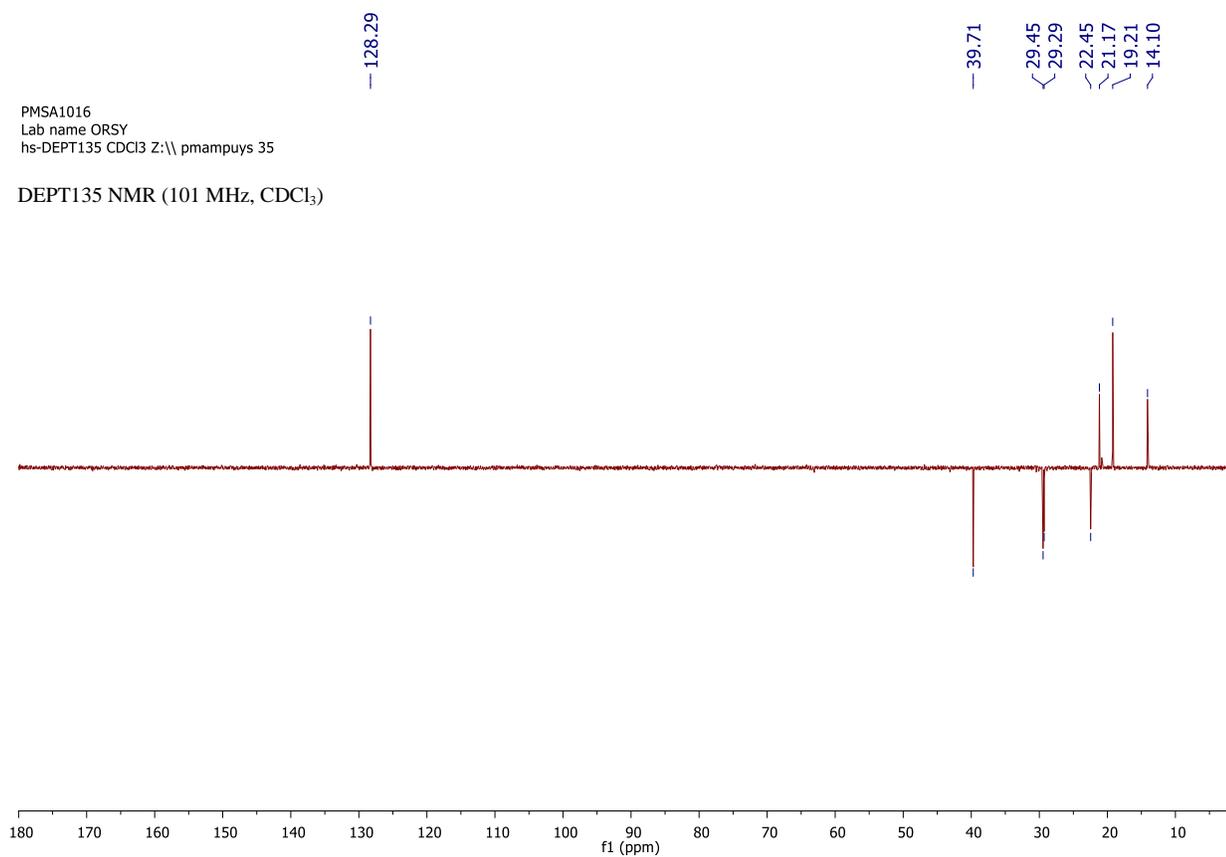
Copies of the NMR spectra



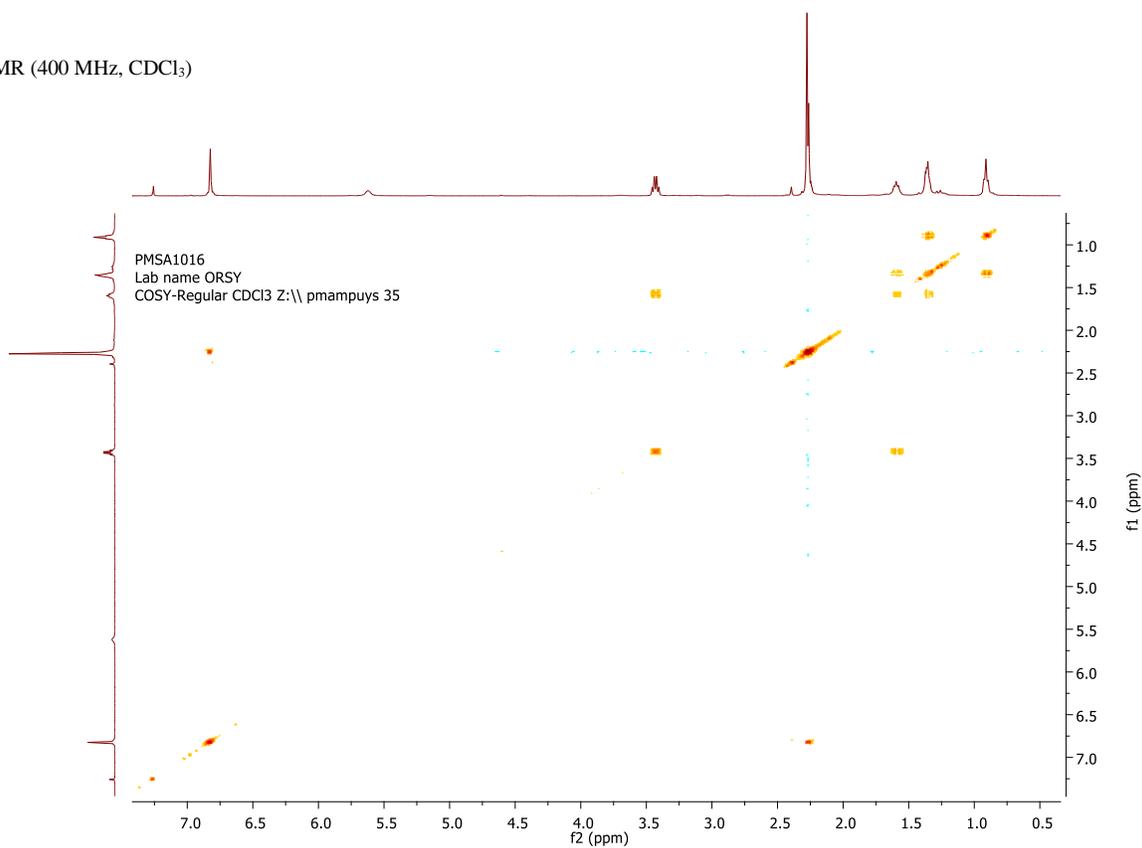
Copies of the NMR spectra

PMSA1016
Lab name ORSY
hs-DEPT135 CDCl₃ Z:\\ pmampuys 35

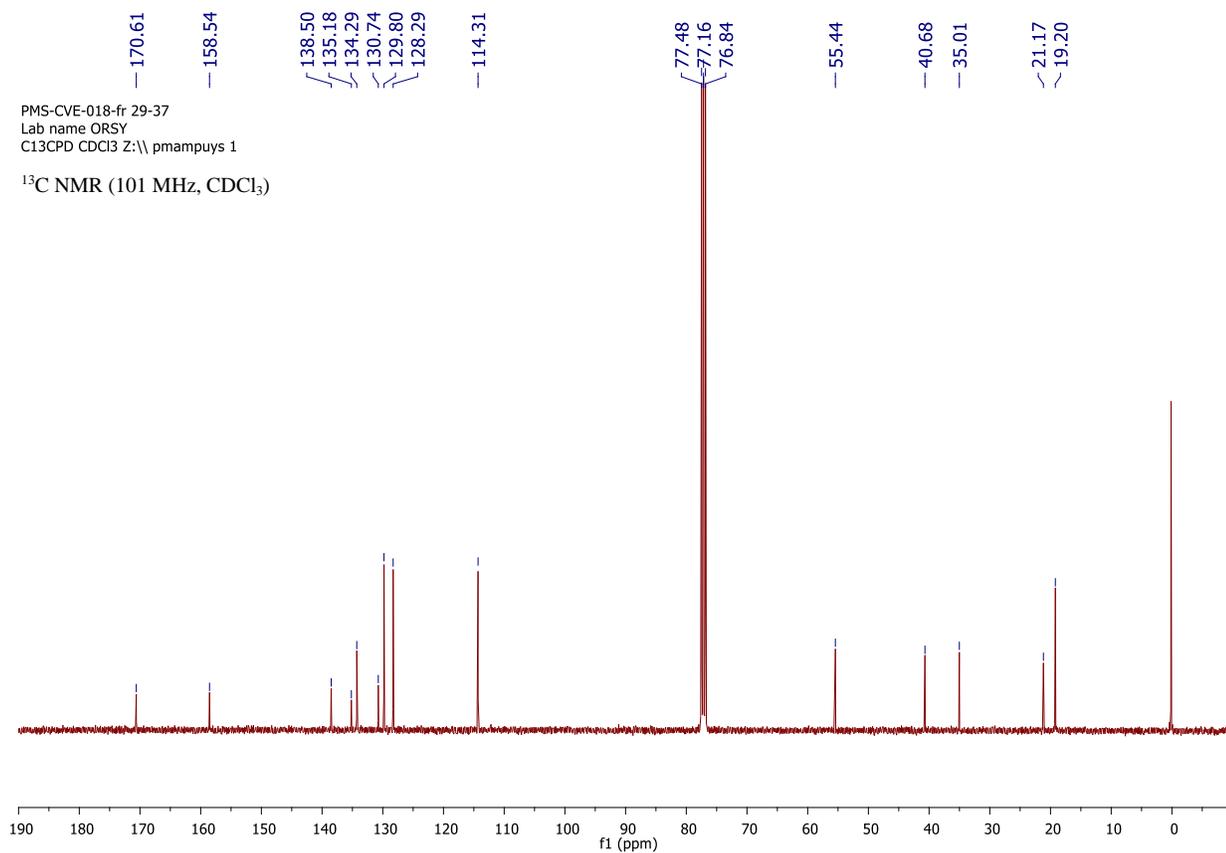
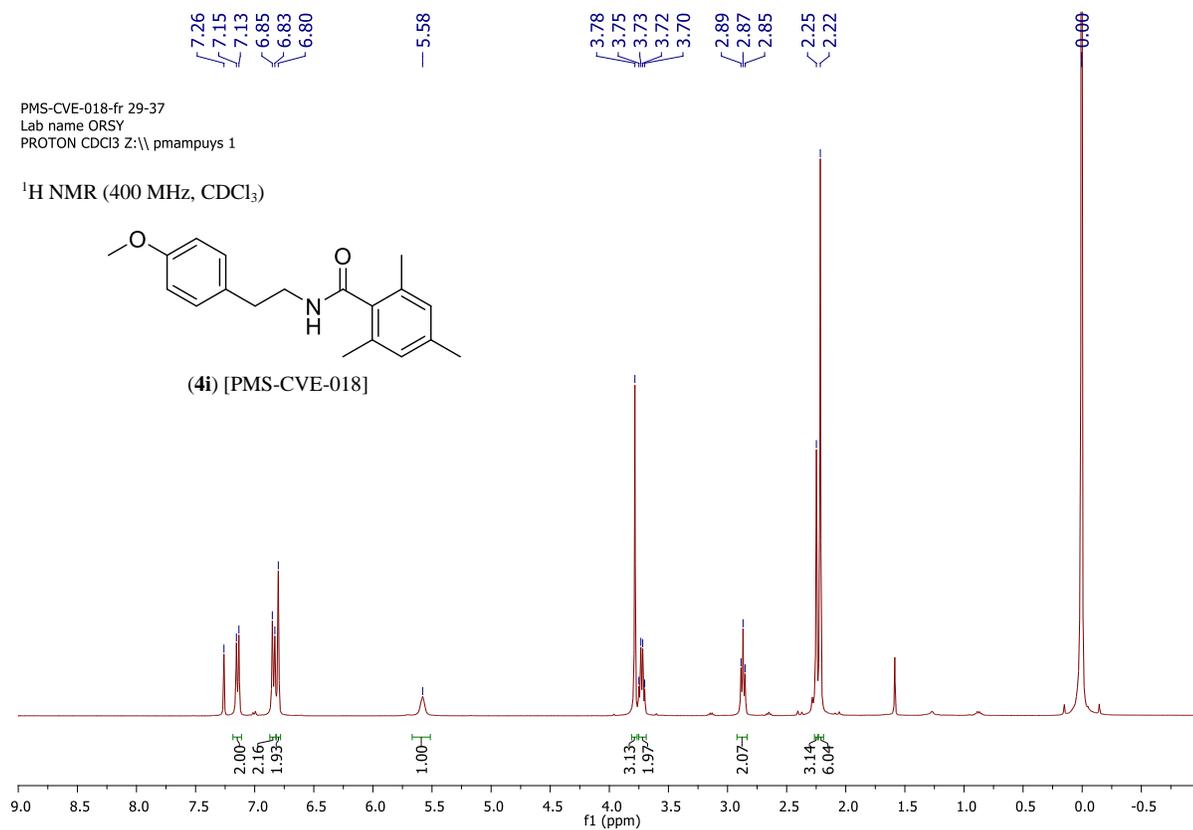
DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)



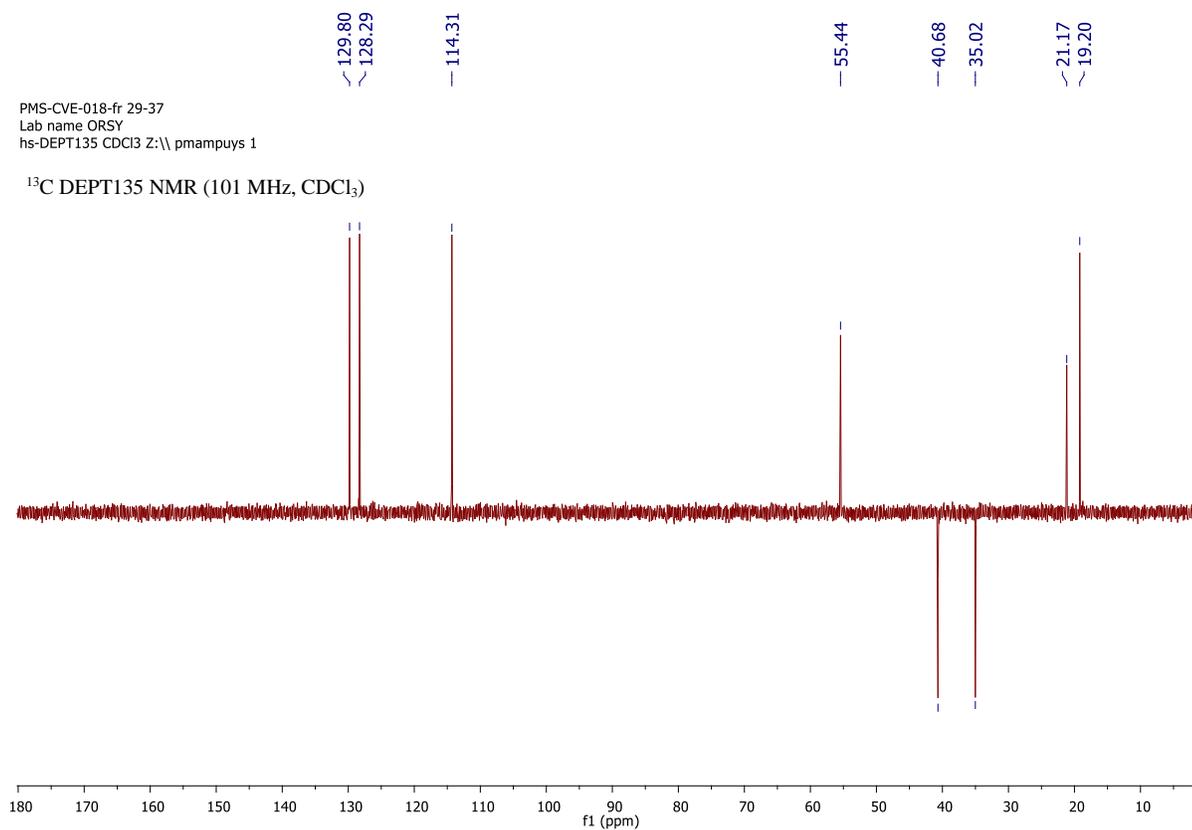
Copies of the NMR spectra



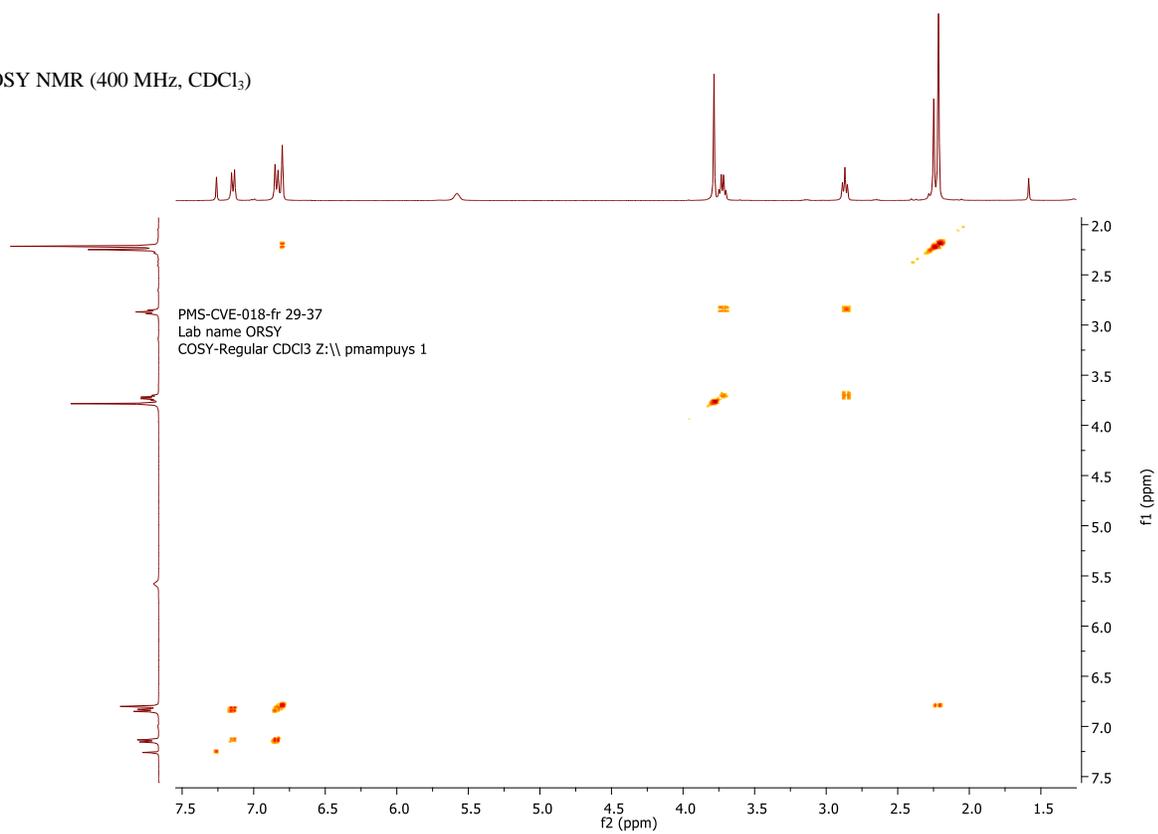
Copies of the NMR spectra

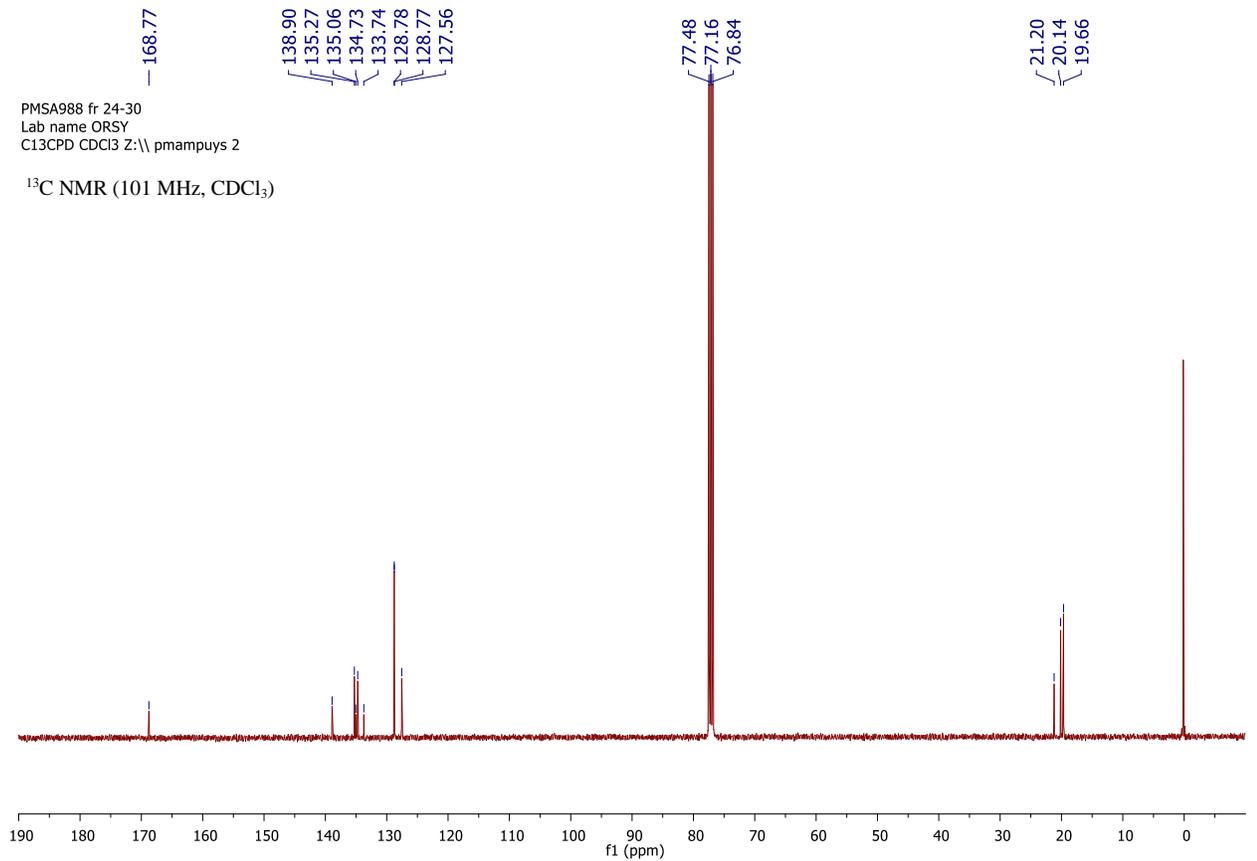
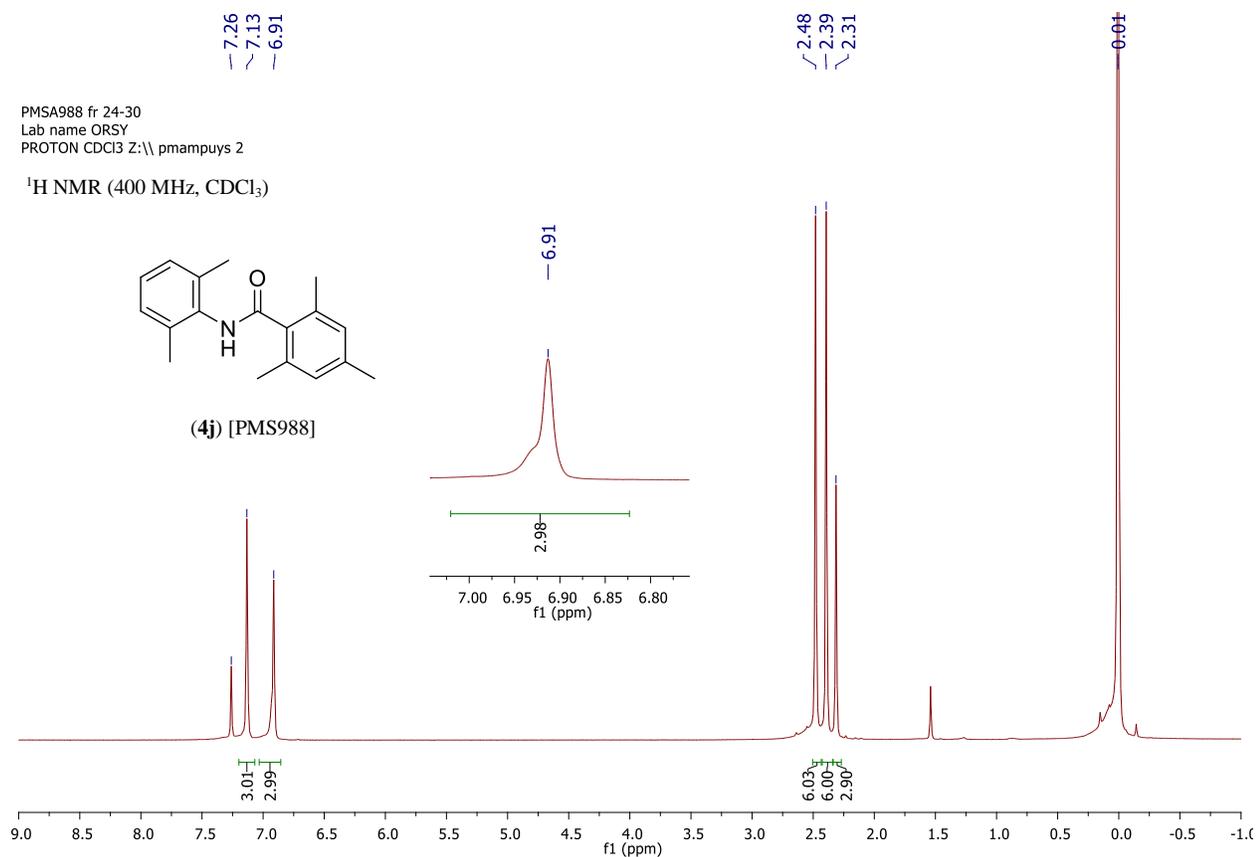
PMS-CVE-018-fr 29-37
Lab name ORSY
hs-DEPT135 CDCl3 Z:\ pmampuys 1

¹³C DEPT135 NMR (101 MHz, CDCl₃)



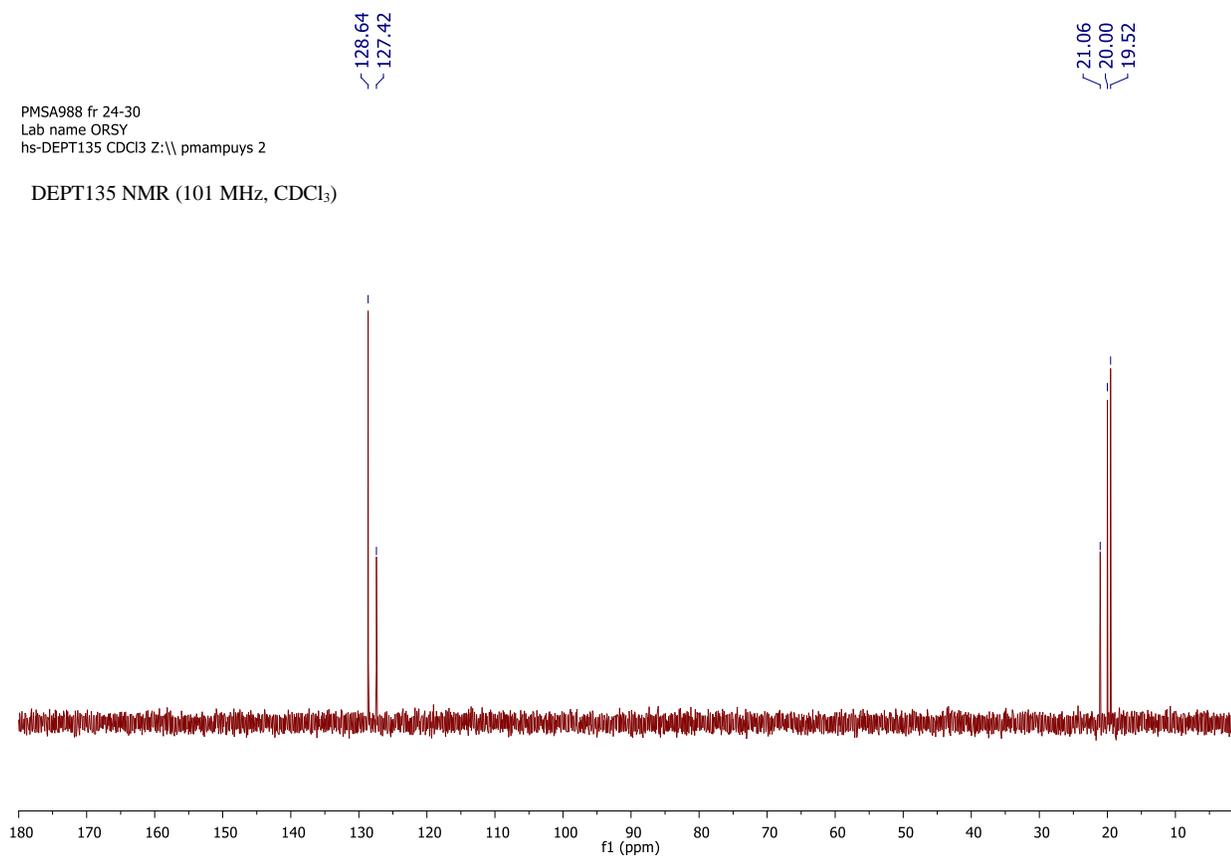
COSY NMR (400 MHz, CDCl₃)



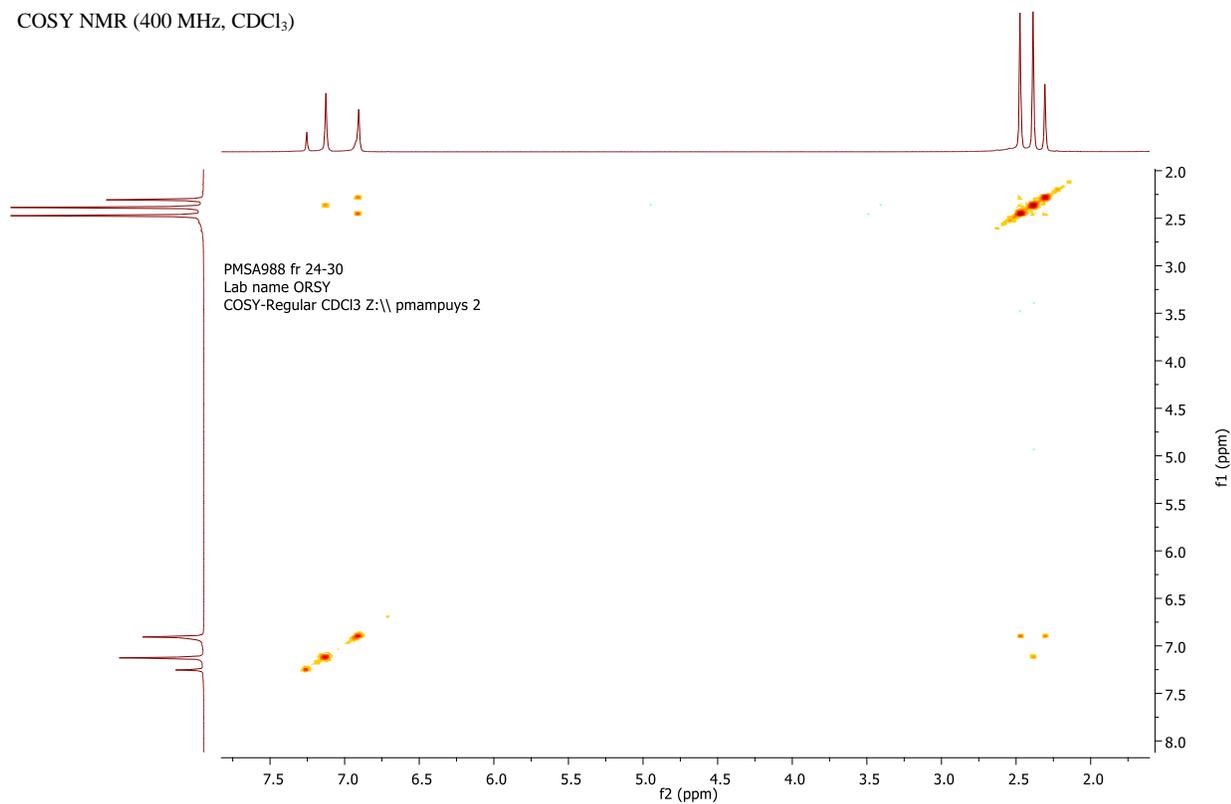


PMSA988 fr 24-30
Lab name ORSY
hs-DEPT135 CDCl3 Z:\\ pmampuy 2

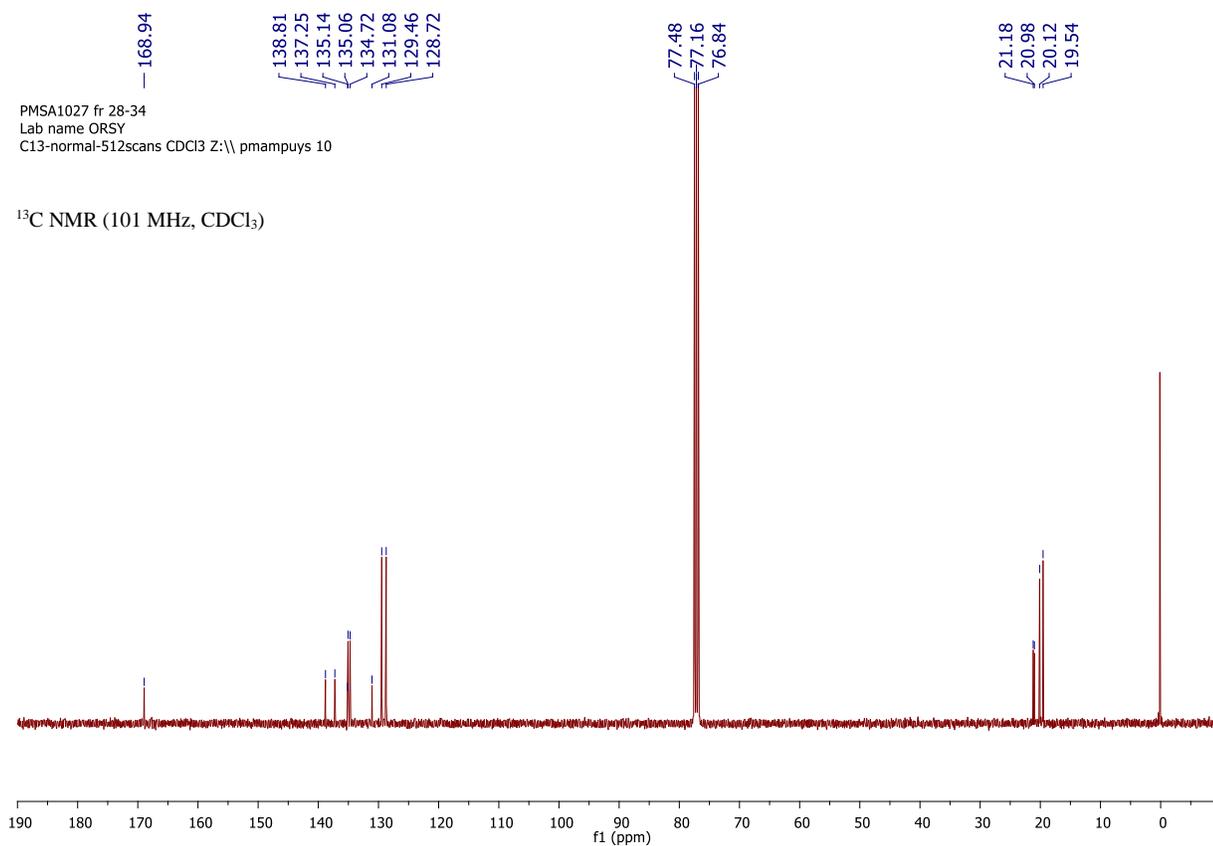
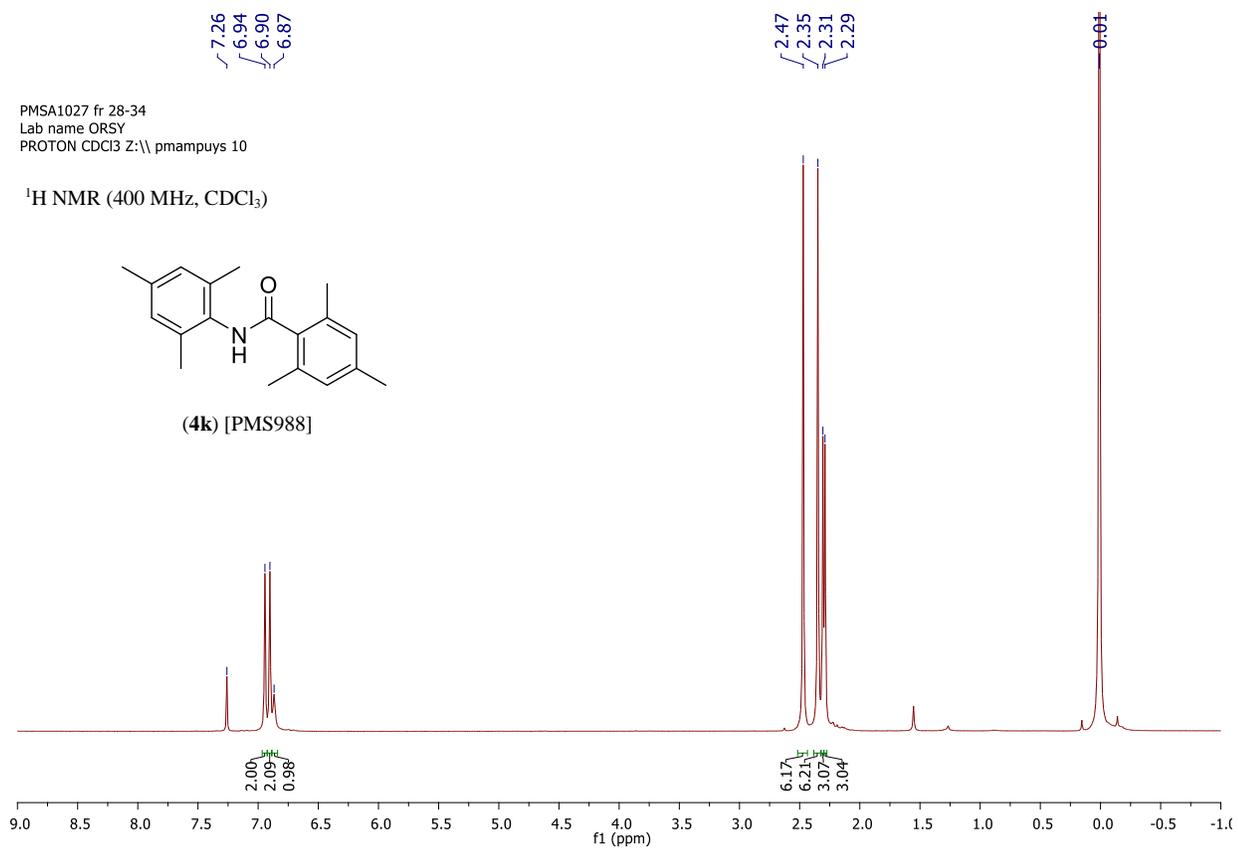
DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)



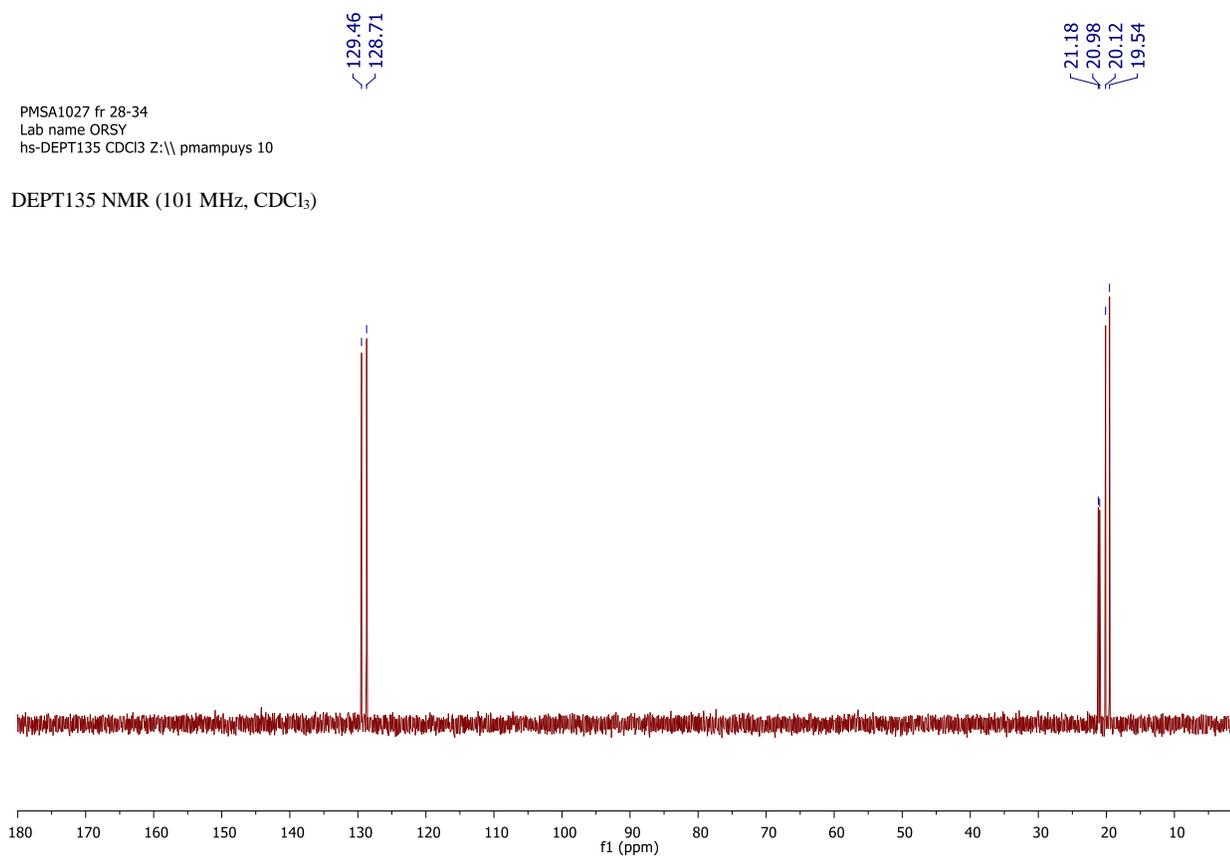
PMSA988 fr 24-30
Lab name ORSY
COSY-Regular CDCl3 Z:\\ pmampuy 2



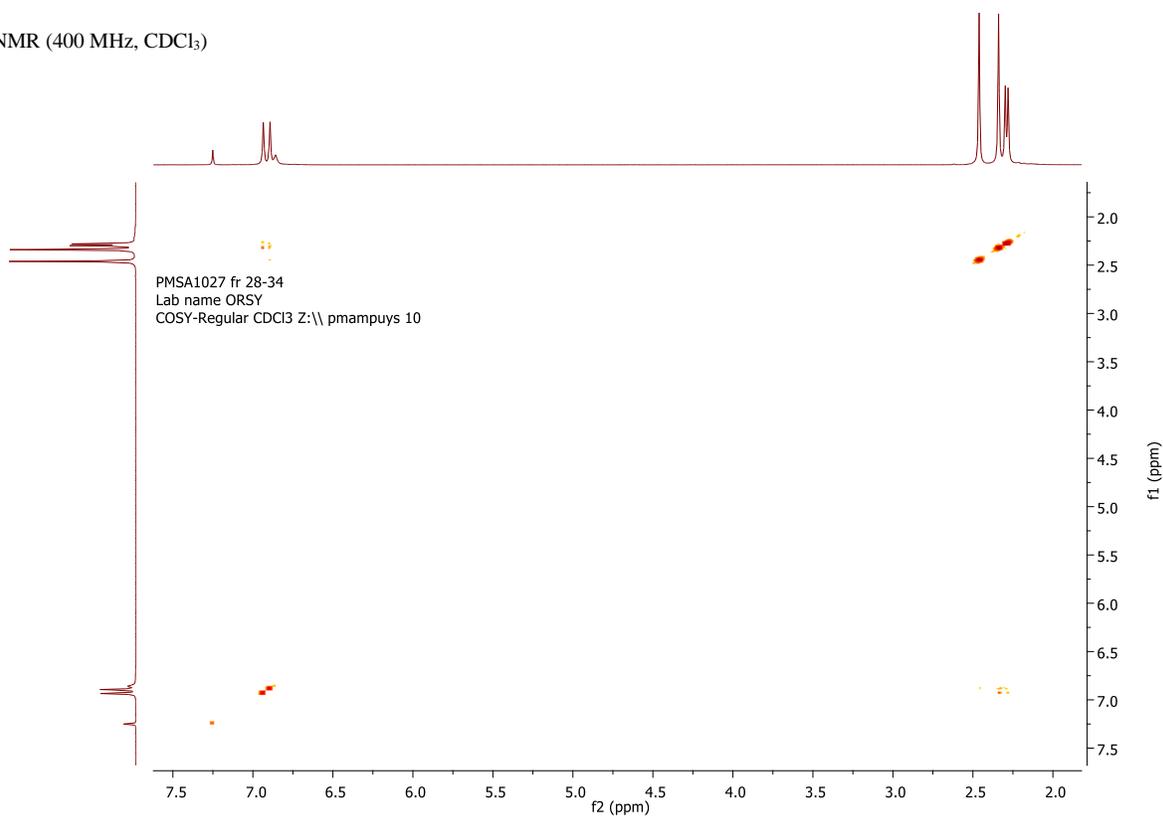
Copies of the NMR spectra

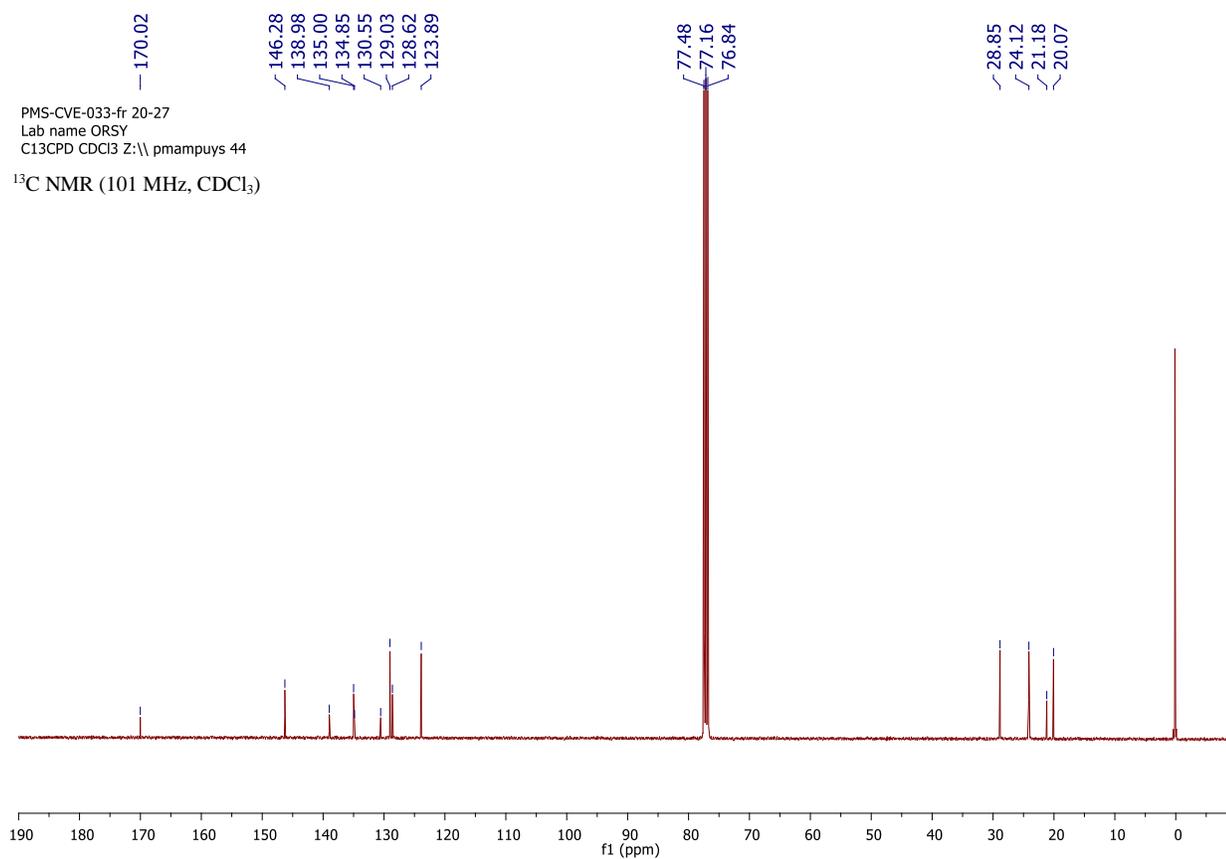
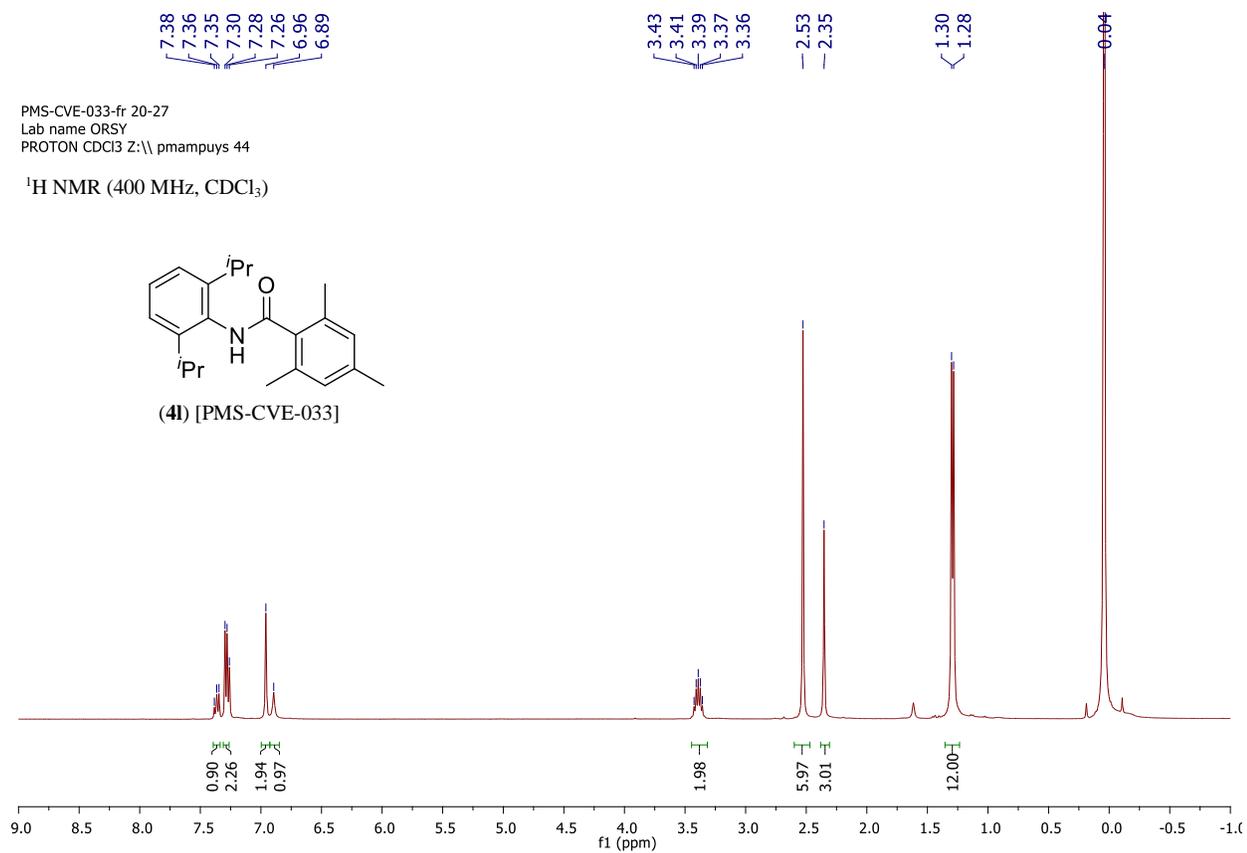
PMSA1027 fr 28-34
Lab name ORSY
hs-DEPT135 CDCl₃ Z:\\ pmampuys 10

DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)



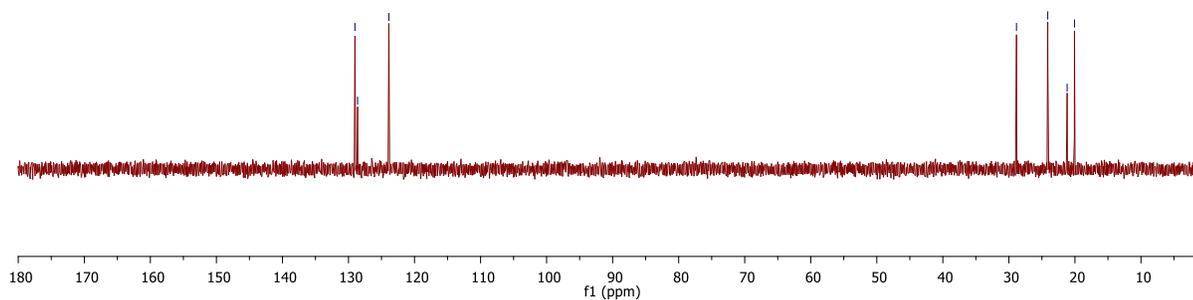


PMS-CVE-033-fr 20-27
Lab name ORSY
hs-DEPT135 CDCl3 Z:\\ pmampuys 44

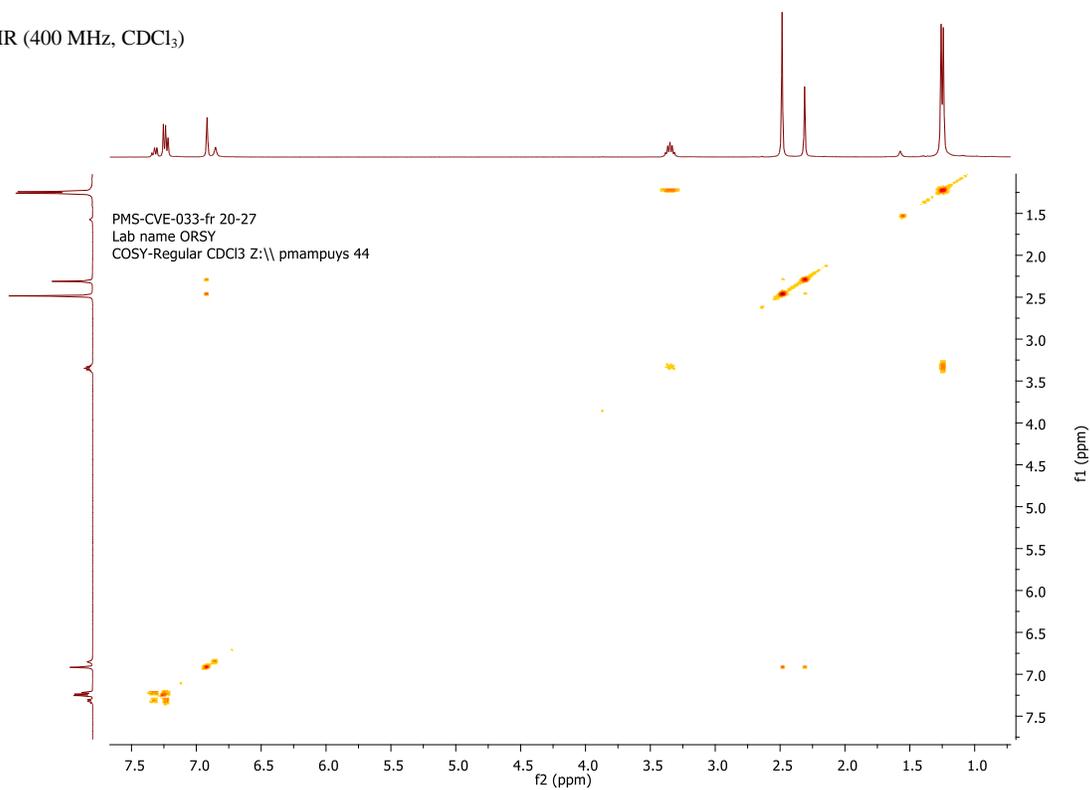
129.02
128.62
123.89

28.85
24.11
21.18
20.07

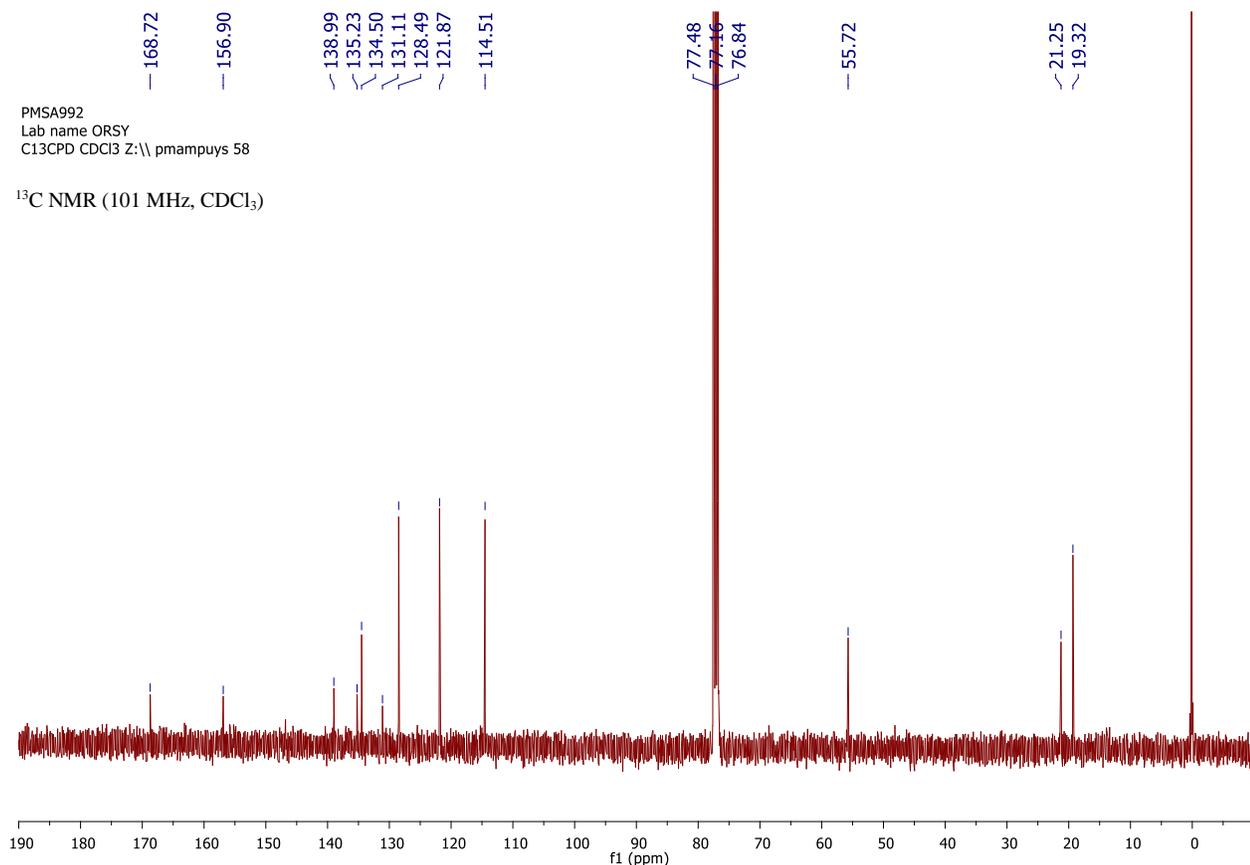
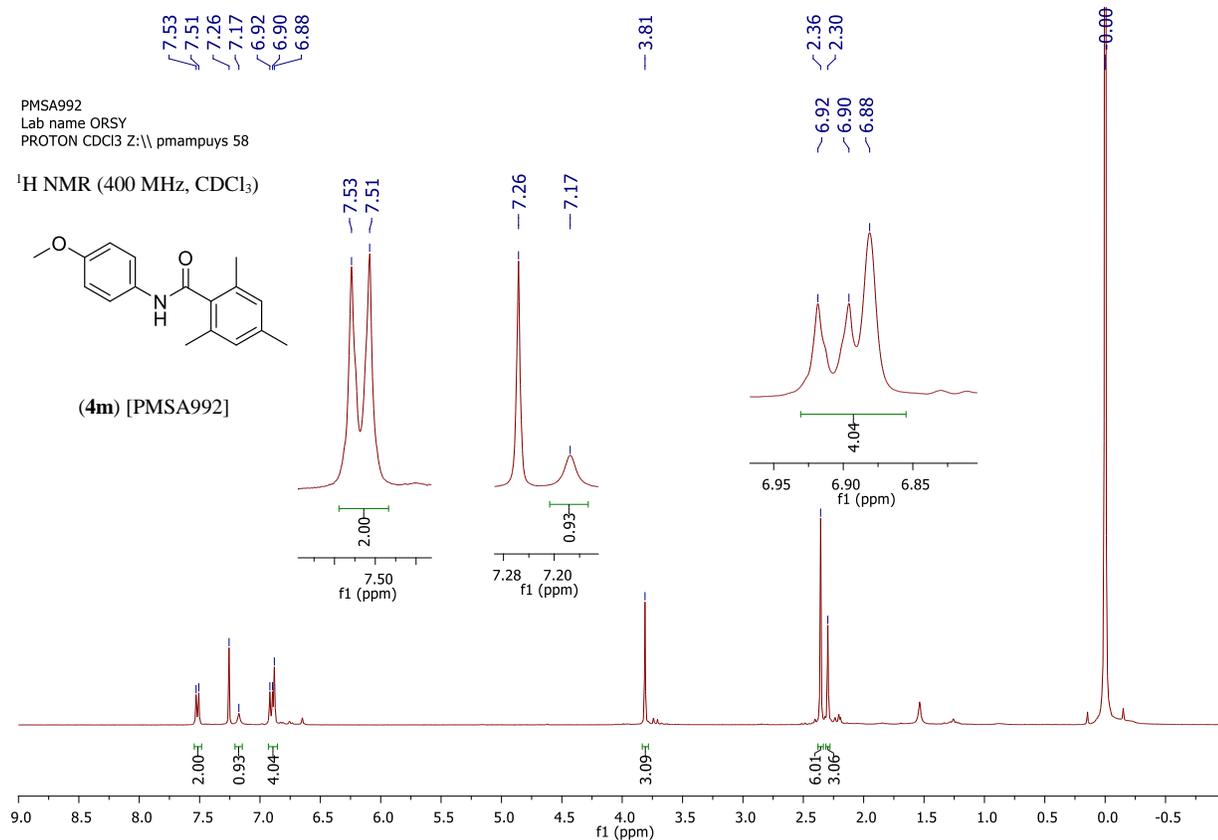
DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)



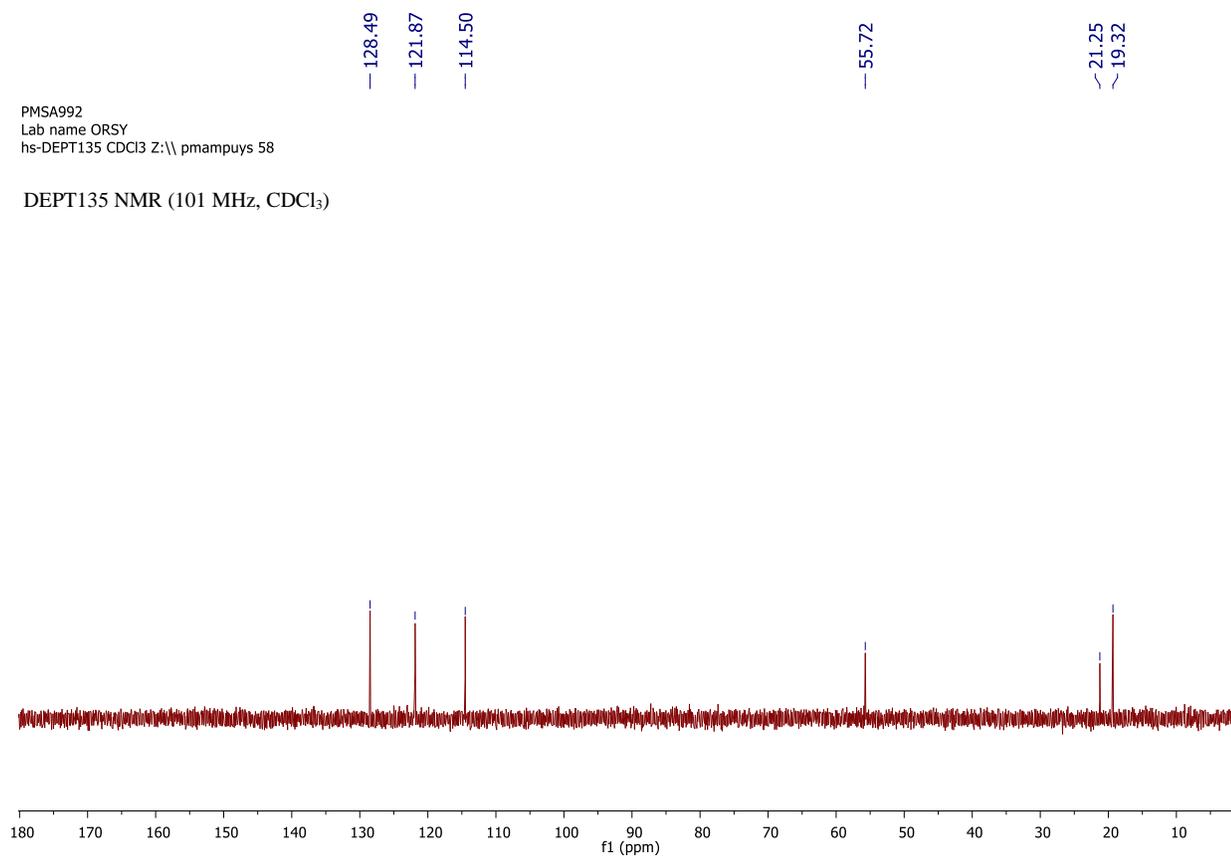
Copies of the NMR spectra



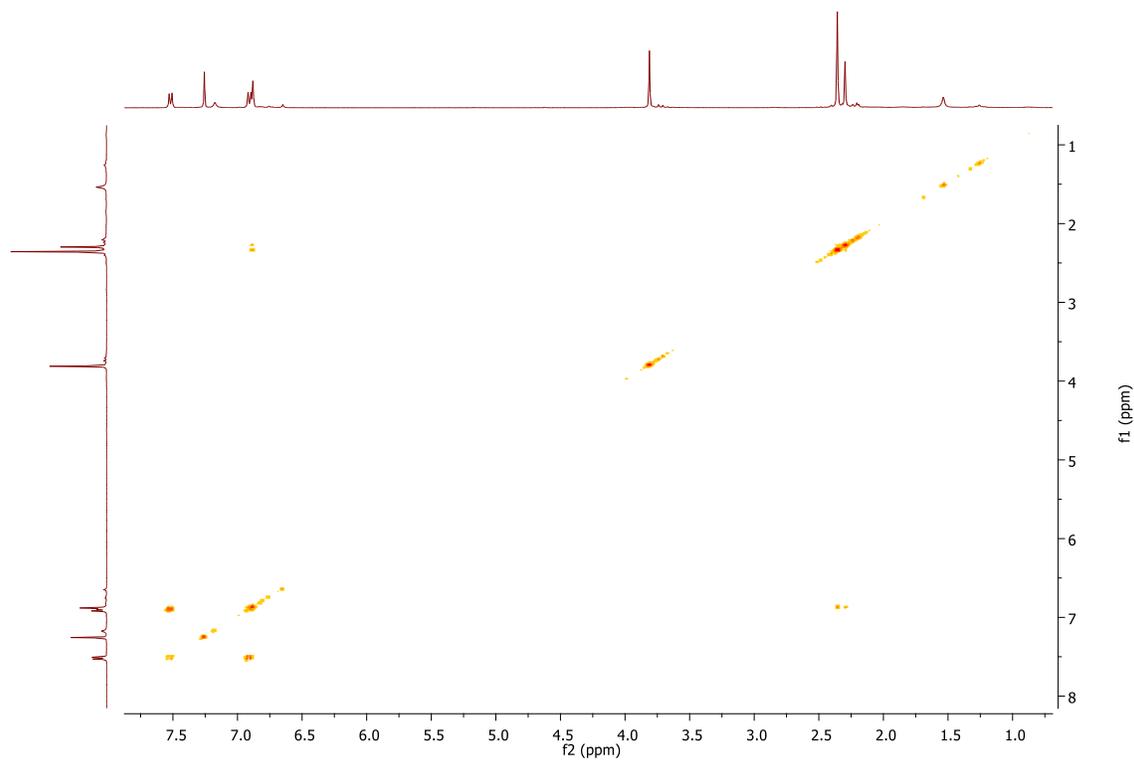
Copies of the NMR spectra

PMSA992
Lab name ORSY
hs-DEPT135 CDCl3 Z:\\ pmampuy5 58

DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)

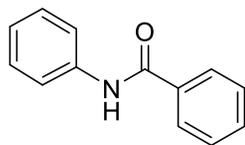


Copies of the NMR spectra

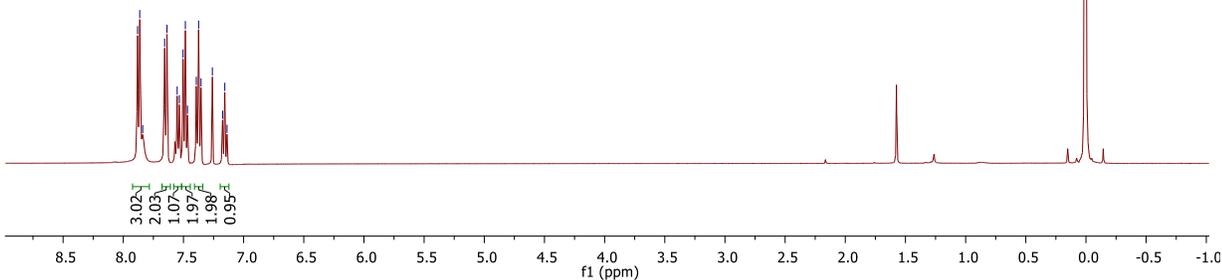
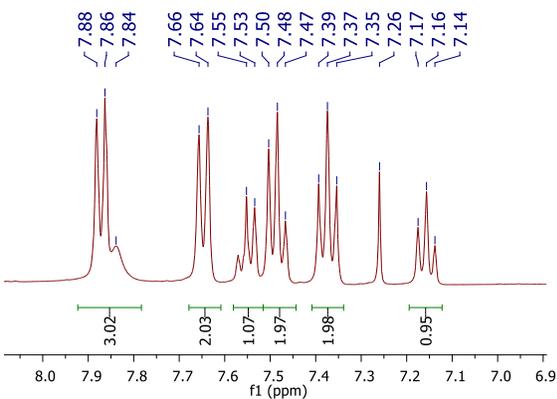
7.88
7.86
7.84
7.66
7.64
7.55
7.53
7.50
7.48
7.47
7.39
7.37
7.35
7.26
7.17
7.16
7.14

PMSD953 fr 18-22
Lab name ORSY
PROTON CDCI3 Z:\ pmampuy 26

¹H NMR (400 MHz, CDCl₃)



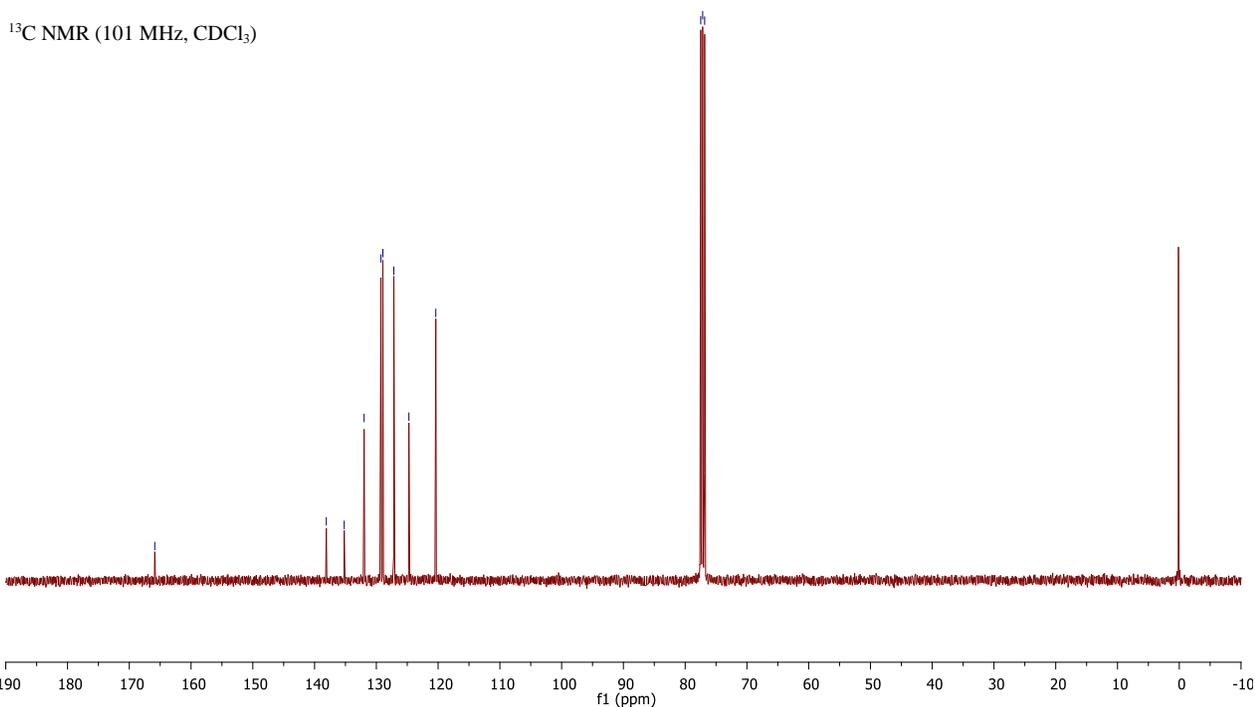
(6a) [PMSA953]



165.88
138.10
135.20
131.98
129.25
128.94
127.17
124.73
120.37

PMSD953 fr 18-22
Lab name ORSY
C13-normal-512scans CDCI3 Z:\ pmampuy 26

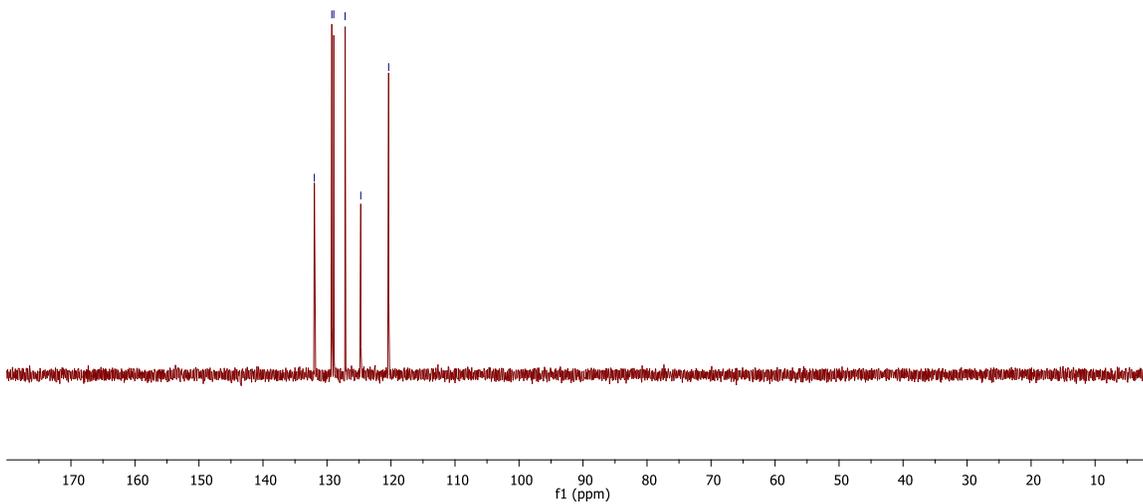
¹³C NMR (101 MHz, CDCl₃)



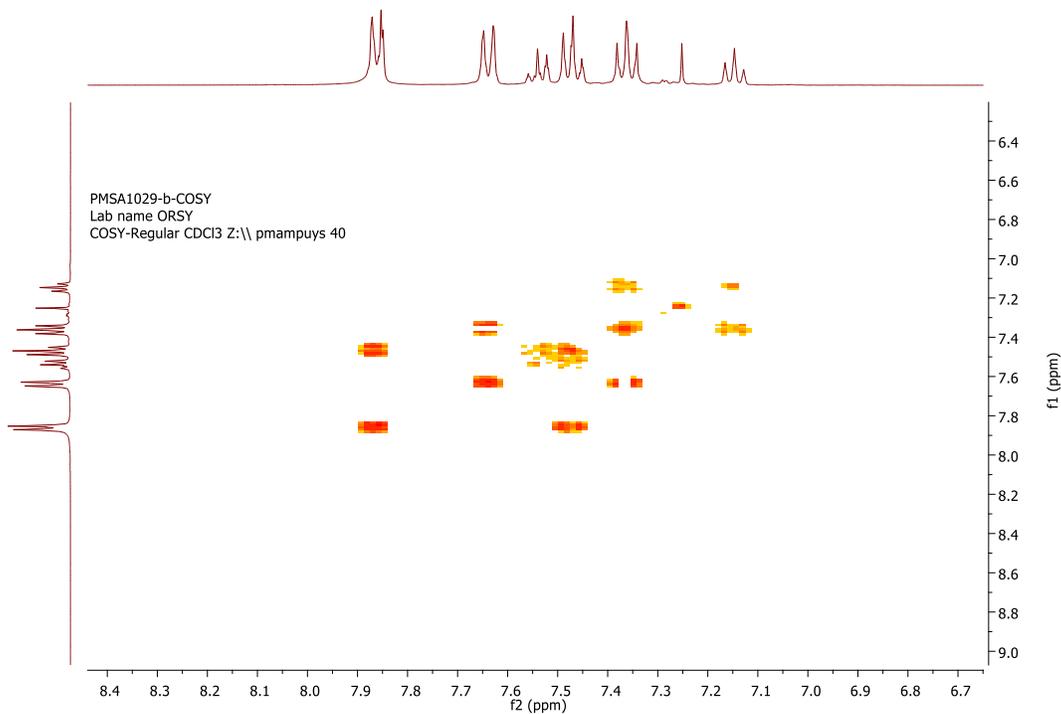
131.97
129.25
128.94
127.16
124.72
120.37

PMSD953 fr 18-22
Lab name ORSY
hs-DEPT135 CDCl₃ Z:\\ pmampuys 26

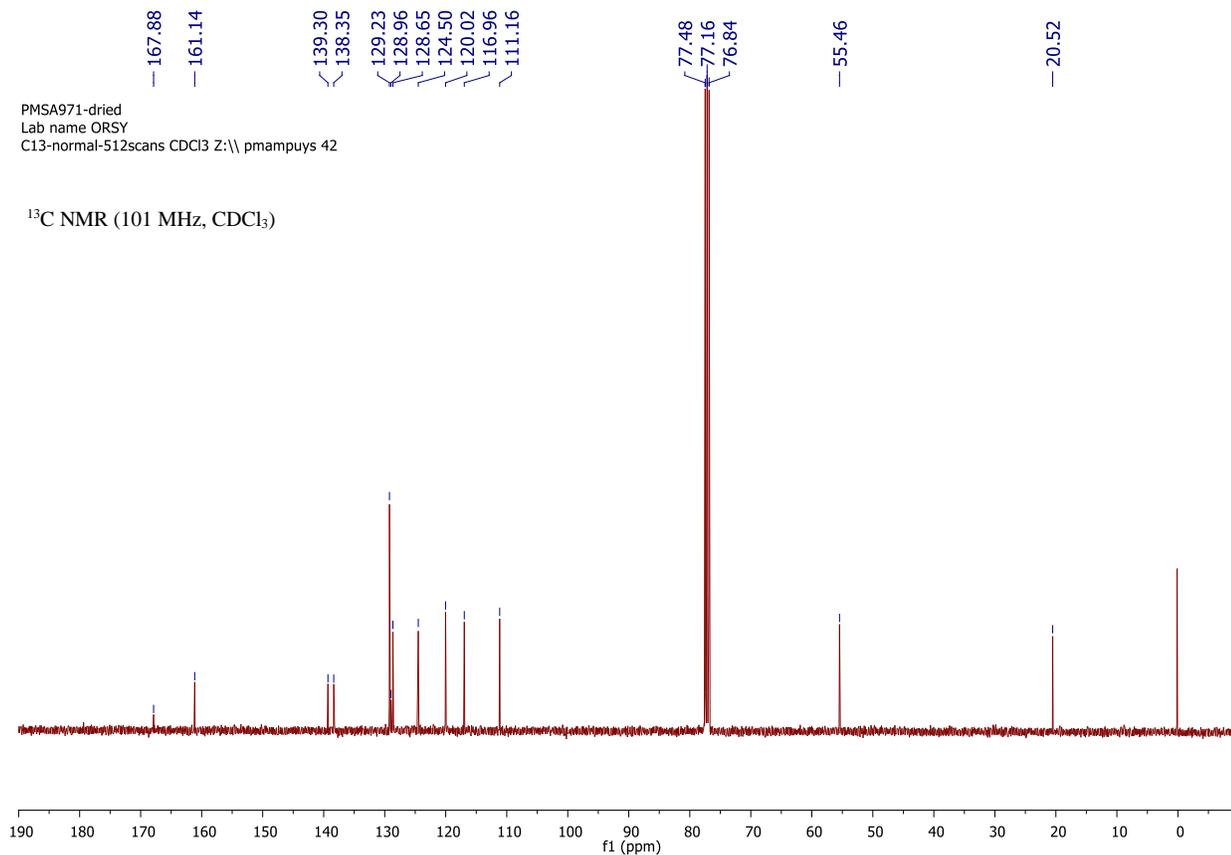
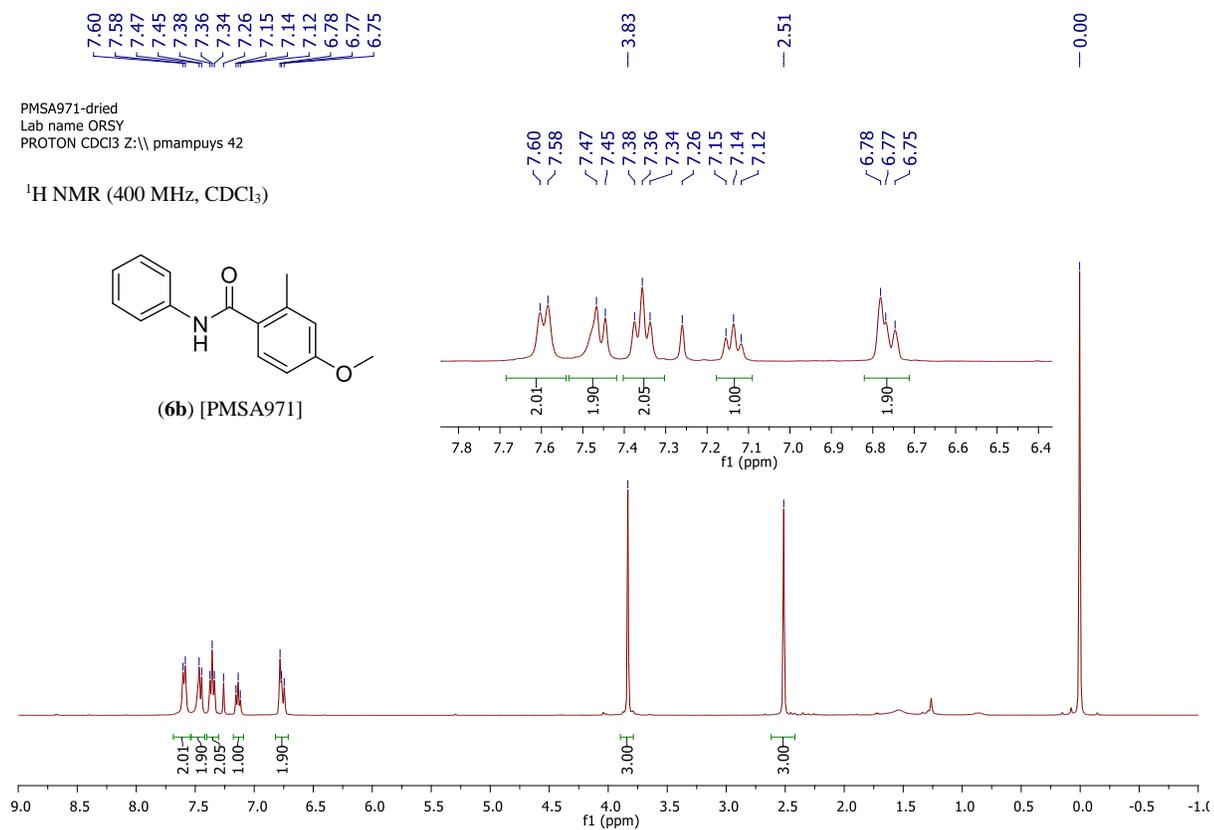
DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)



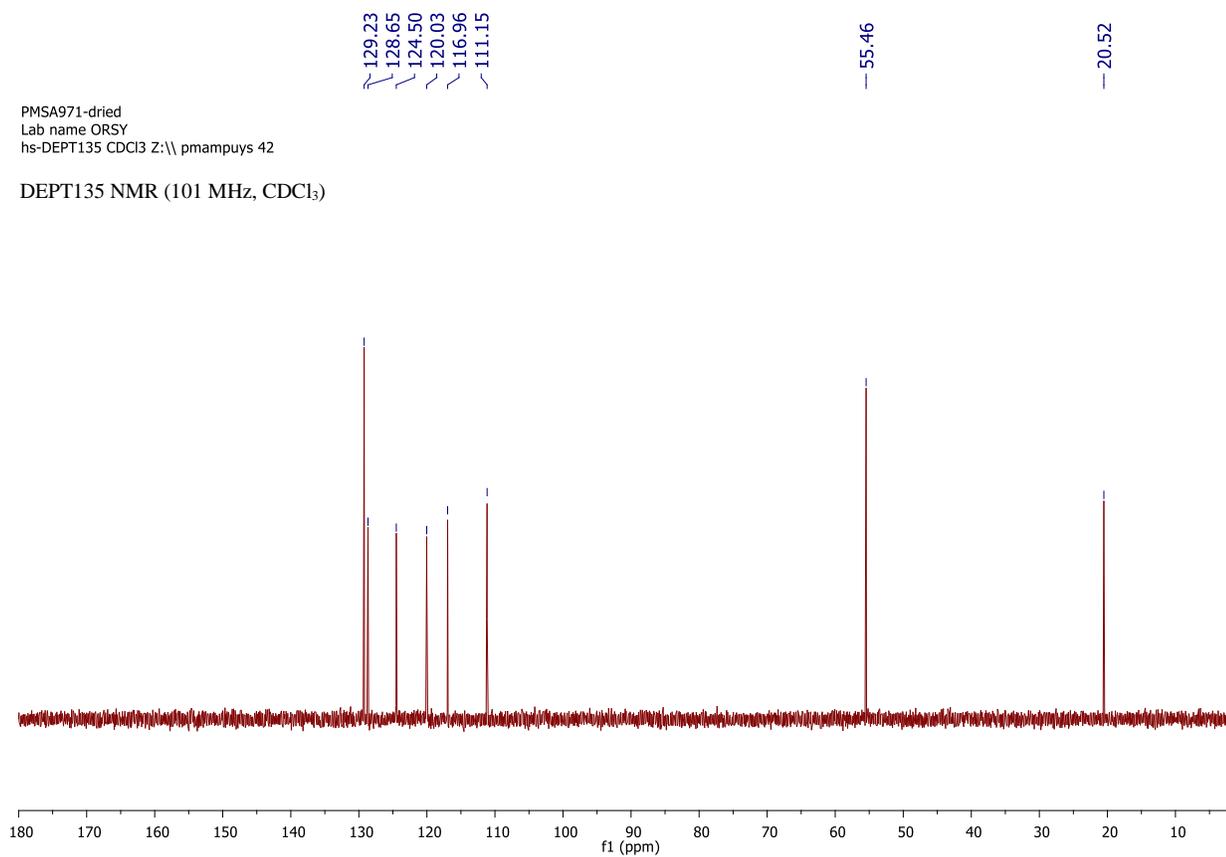
Copies of the NMR spectra



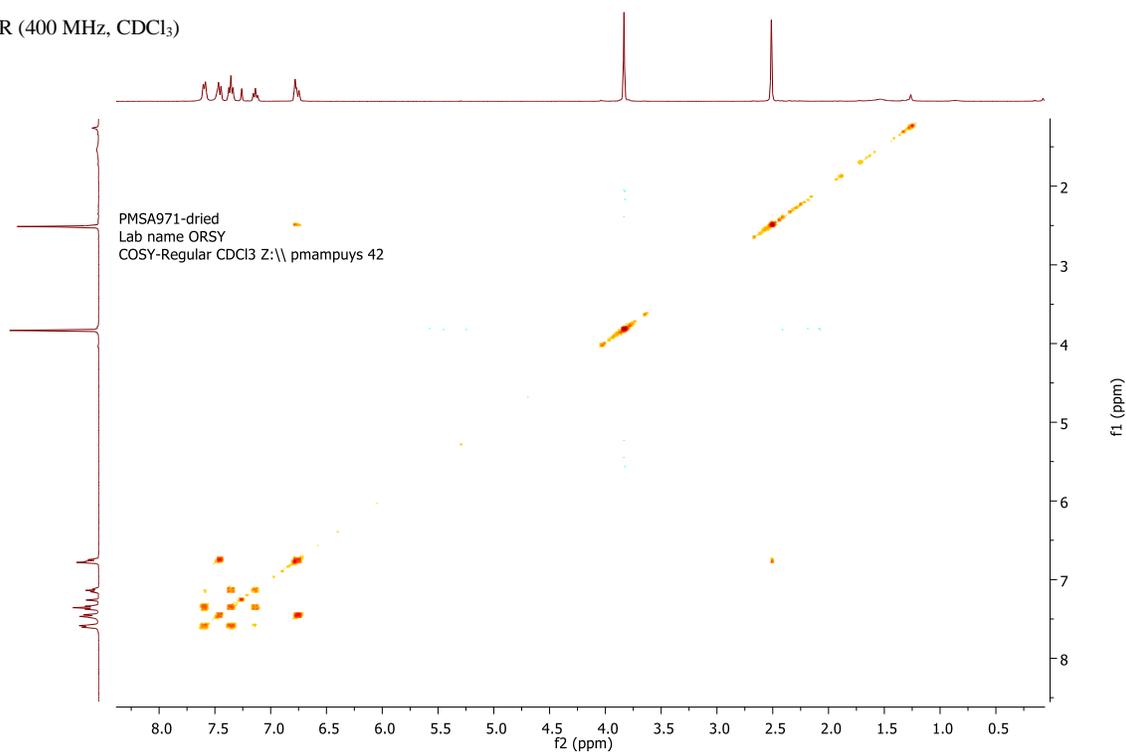
Copies of the NMR spectra

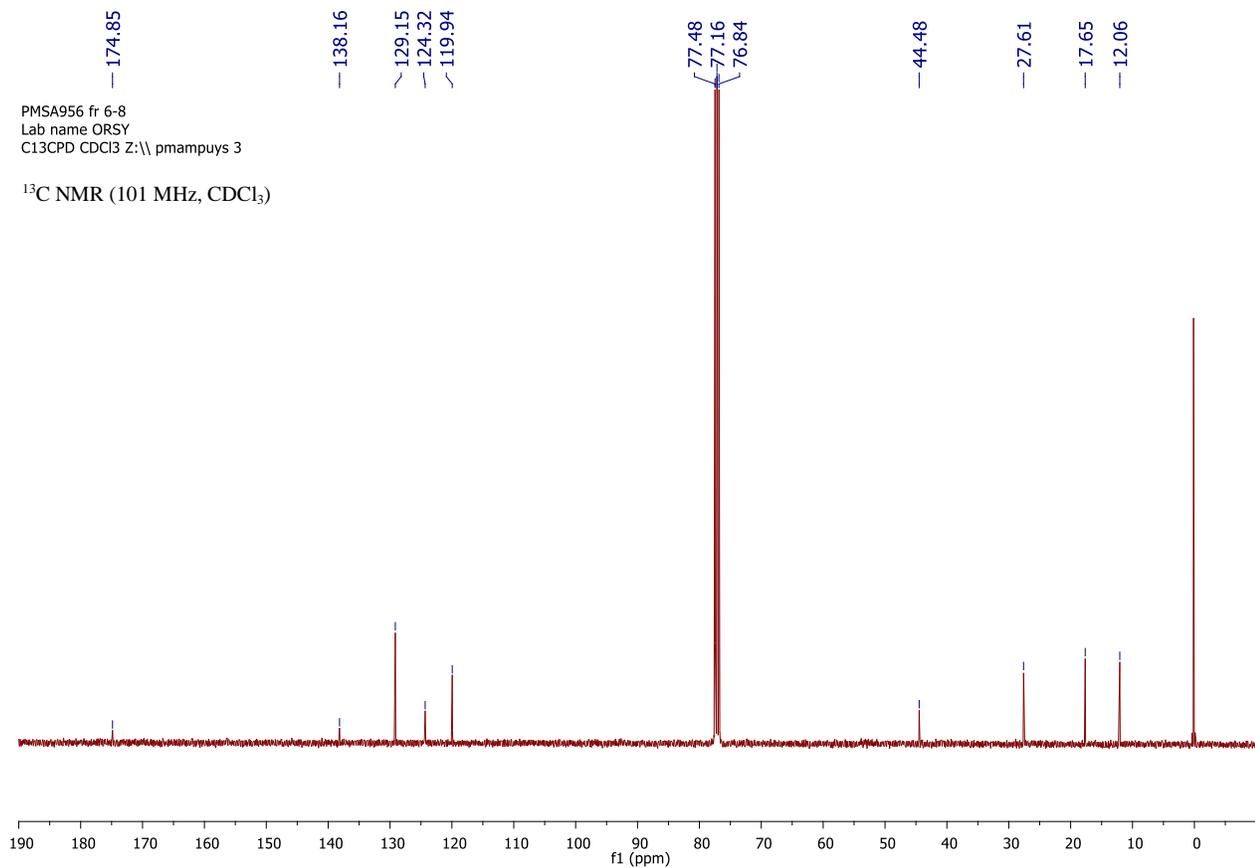
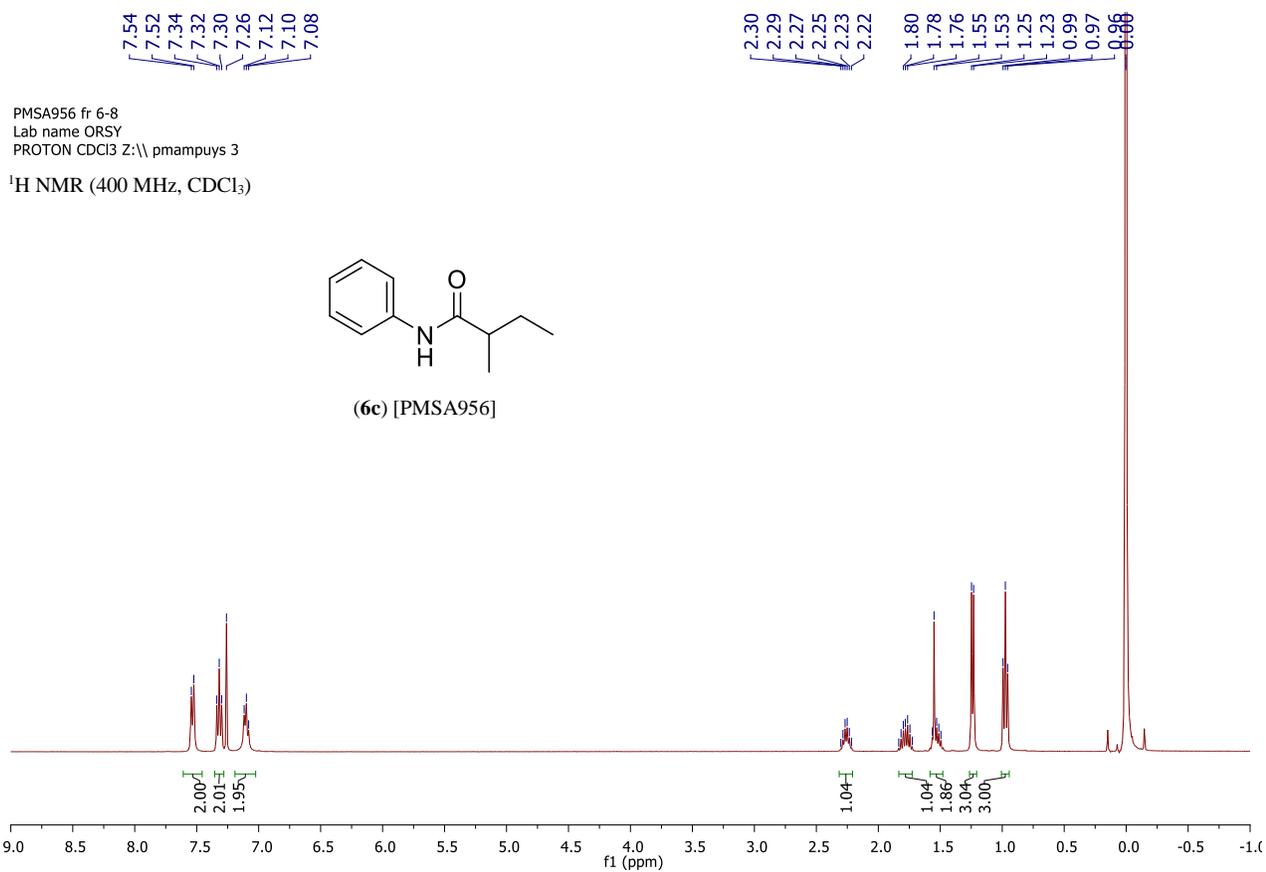
PMSA971-dried
Lab name ORSY
hs-DEPT135 CDCl₃ Z:\\ pmampuys 42

DEPT135 NMR (101 MHz, CDCl₃)



COSY NMR (400 MHz, CDCl₃)

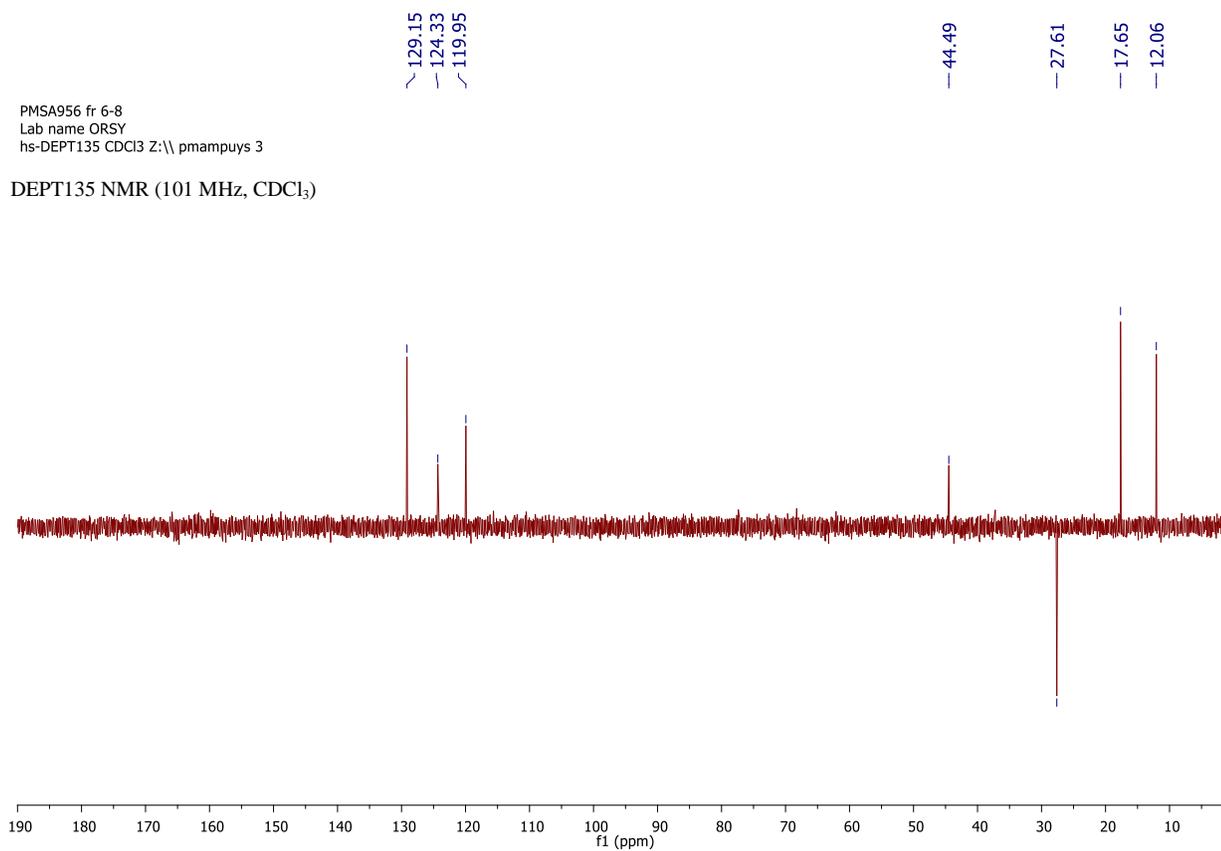




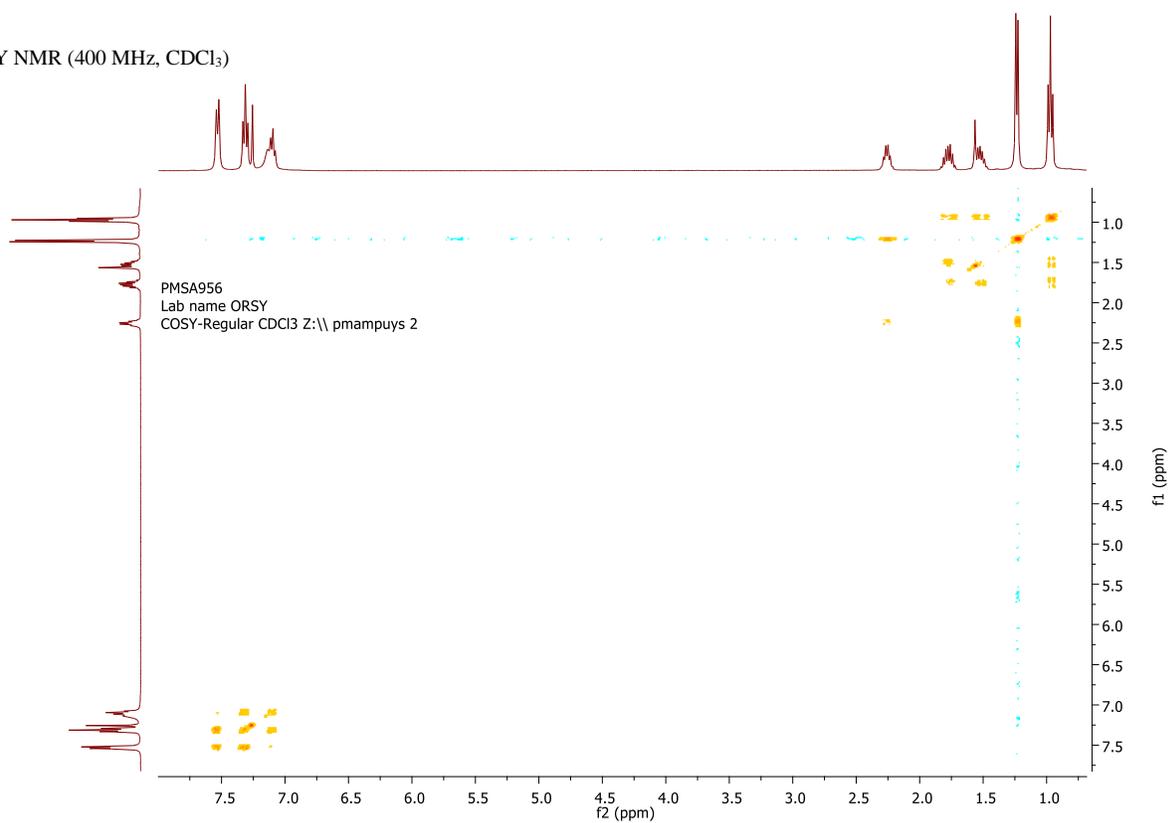
Copies of the NMR spectra

PMSA956 fr 6-8
Lab name ORSY
hs-DEPT135 CDCl3 Z:\\ pmampuys 3

DEPT135 NMR (101 MHz, CDCl₃)



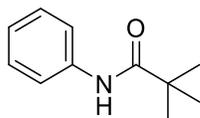
COSY NMR (400 MHz, CDCl₃)



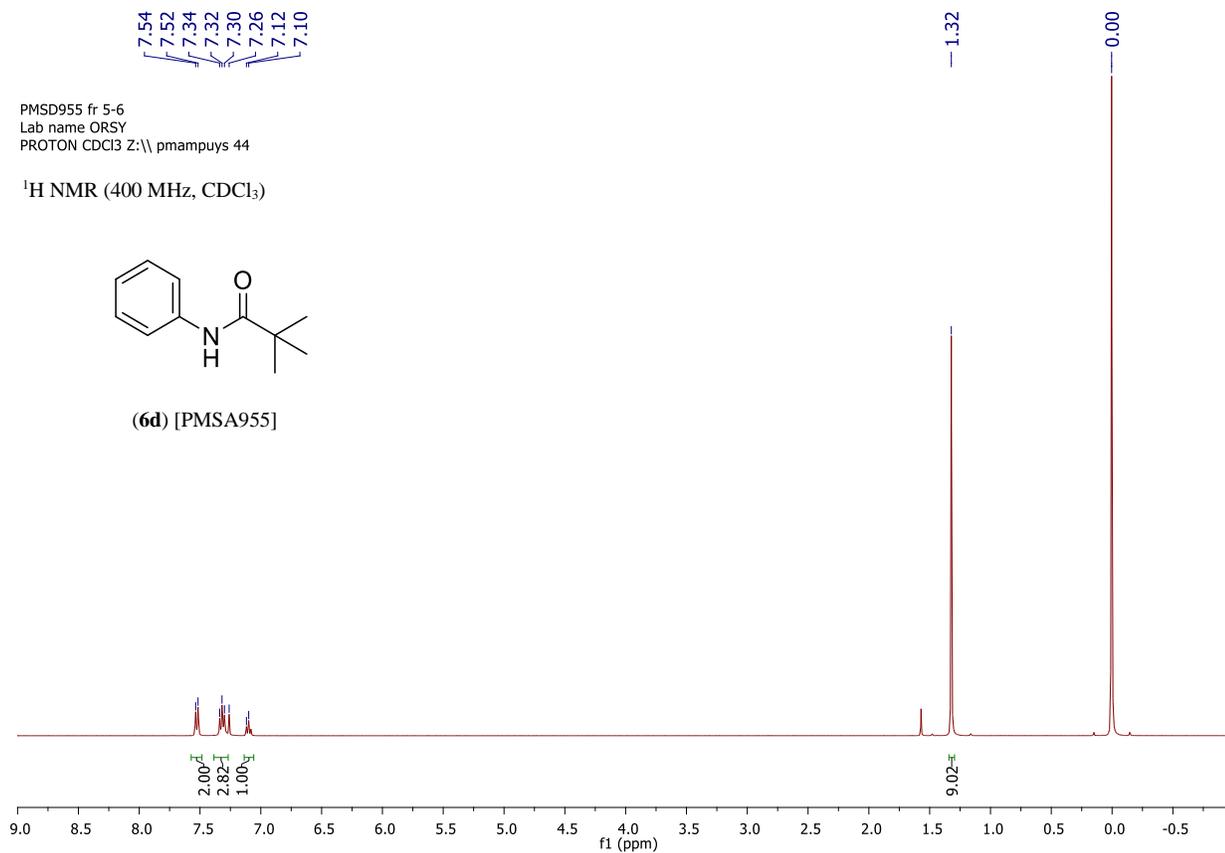
7.54
7.52
7.34
7.32
7.30
7.26
7.12
7.10

PMSD955 fr 5-6
Lab name ORSY
PROTON CDCl3 Z:\ pmampuy5 44

¹H NMR (400 MHz, CDCl₃)



(6d) [PMSA955]



176.66

138.21

129.11

124.34

120.11

77.48

77.16

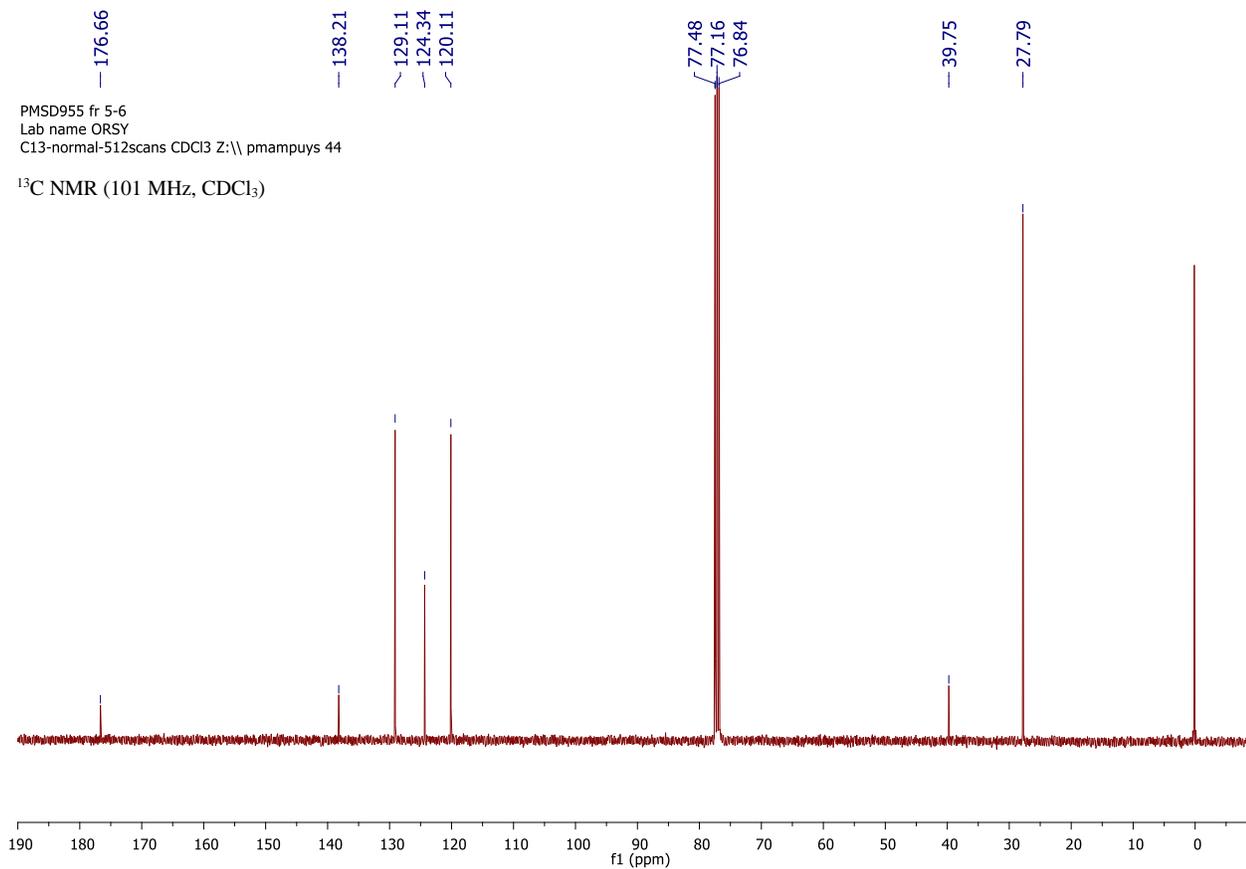
76.84

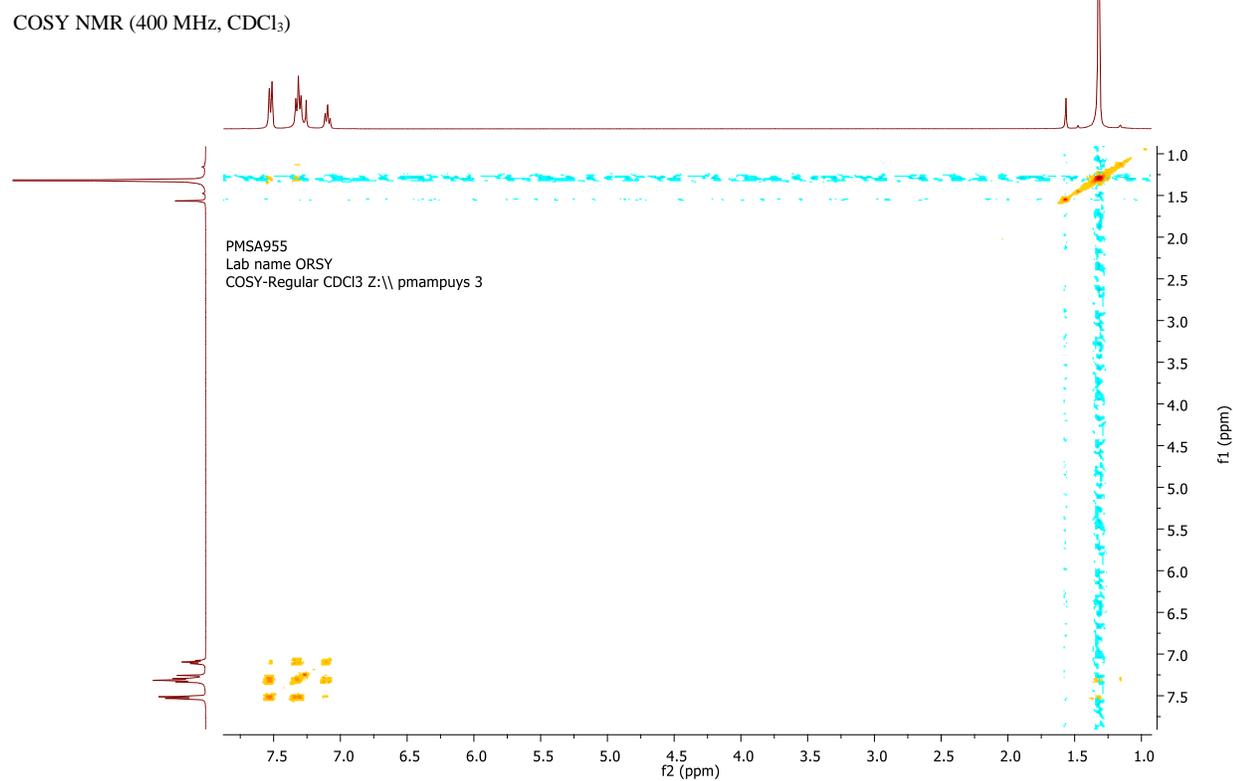
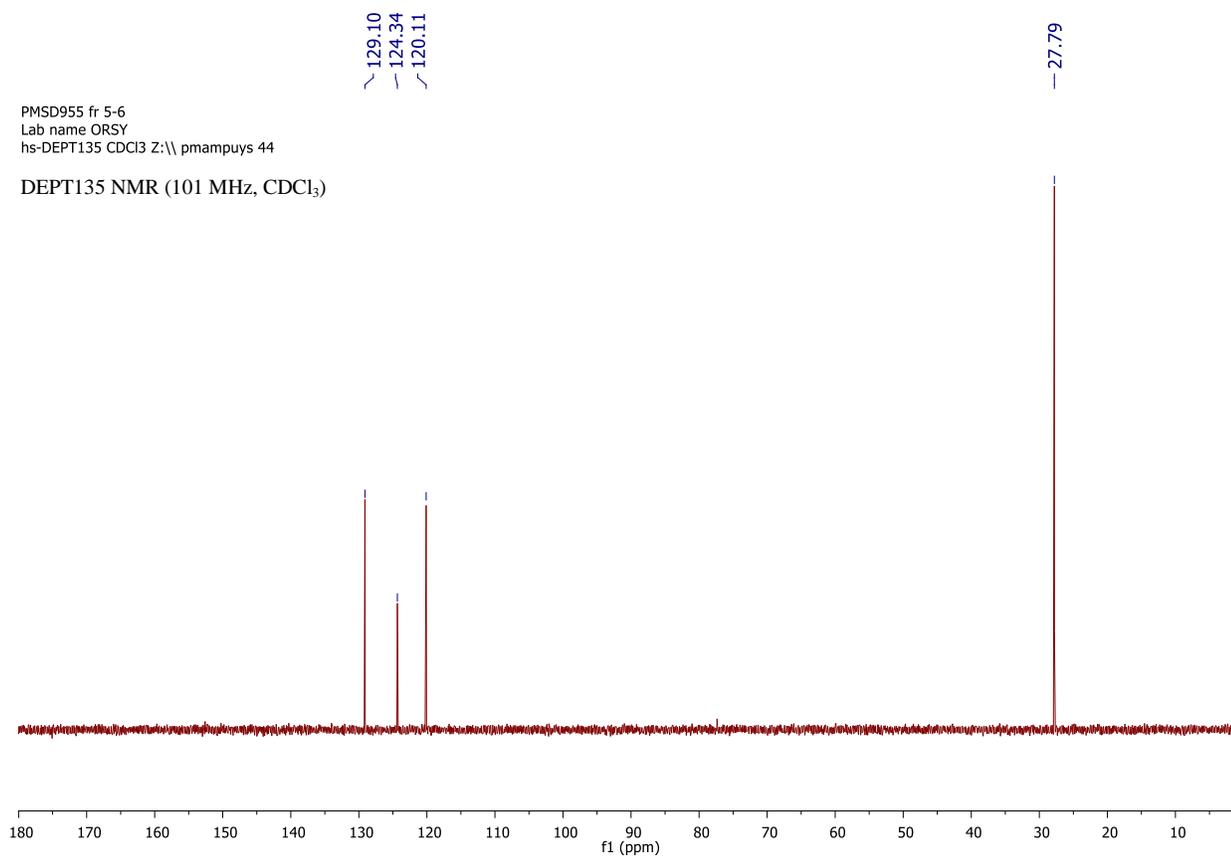
39.75

27.79

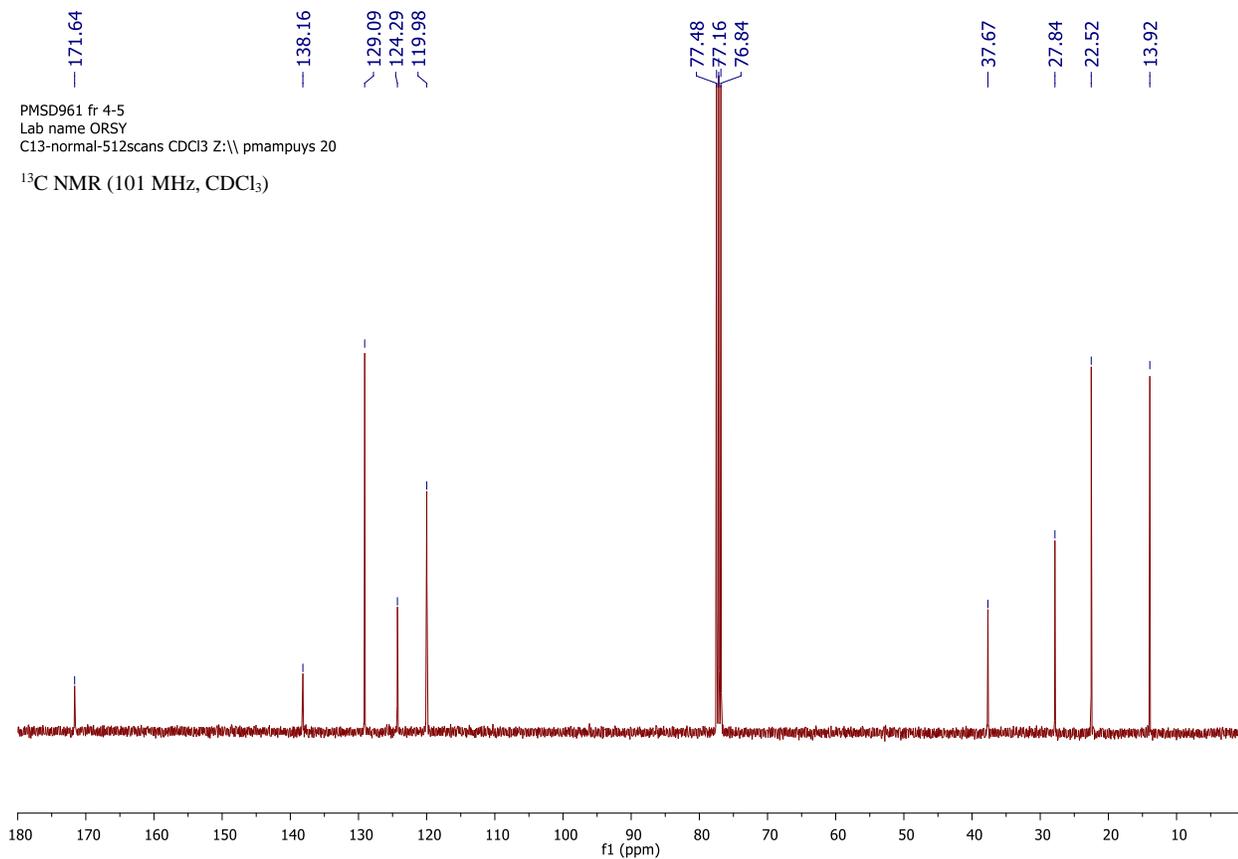
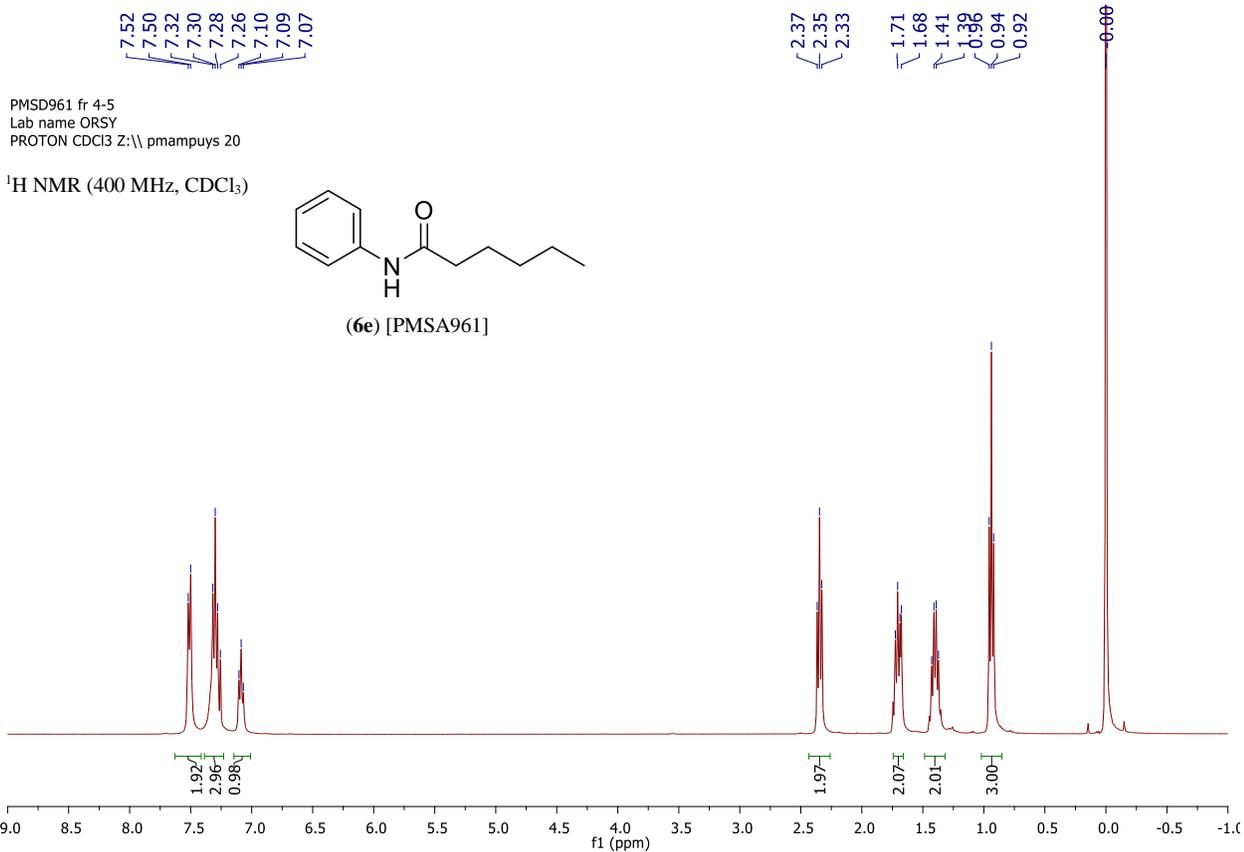
PMSD955 fr 5-6
Lab name ORSY
C13-normal-512scans CDCl3 Z:\ pmampuy5 44

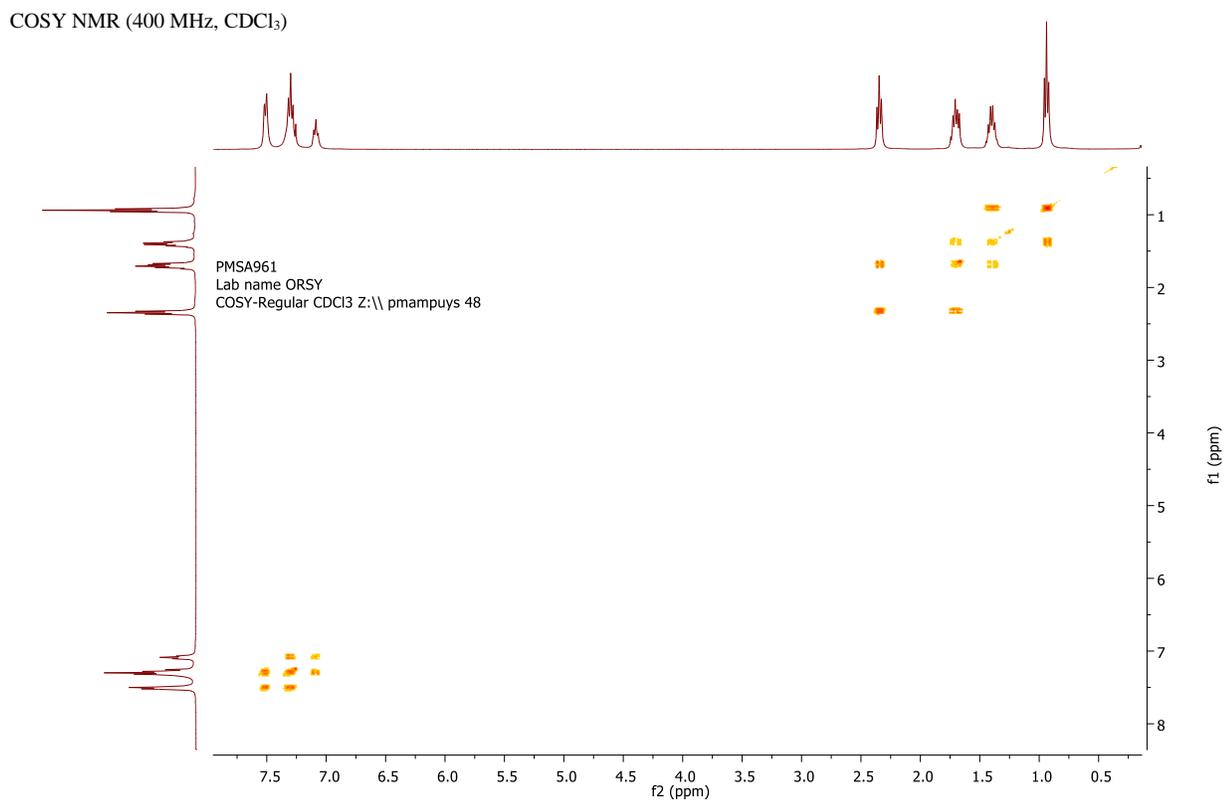
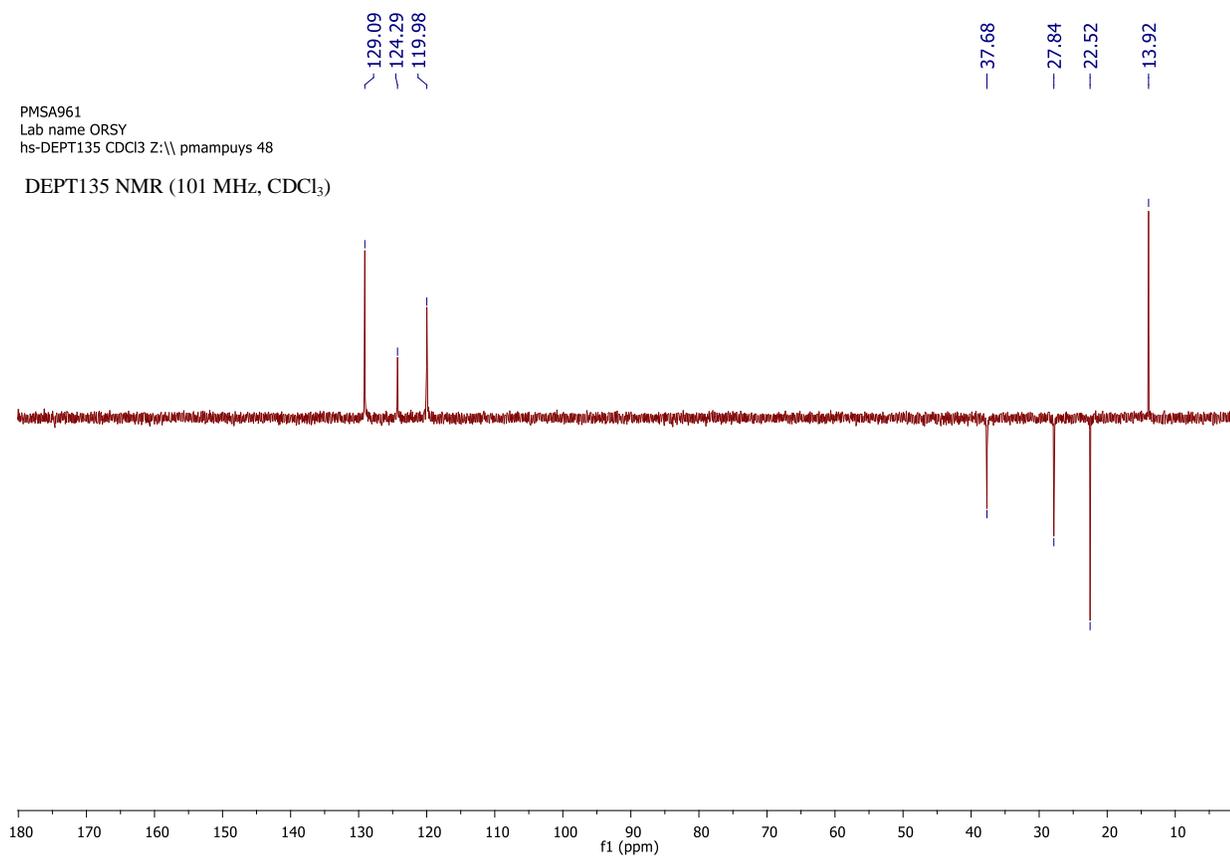
¹³C NMR (101 MHz, CDCl₃)



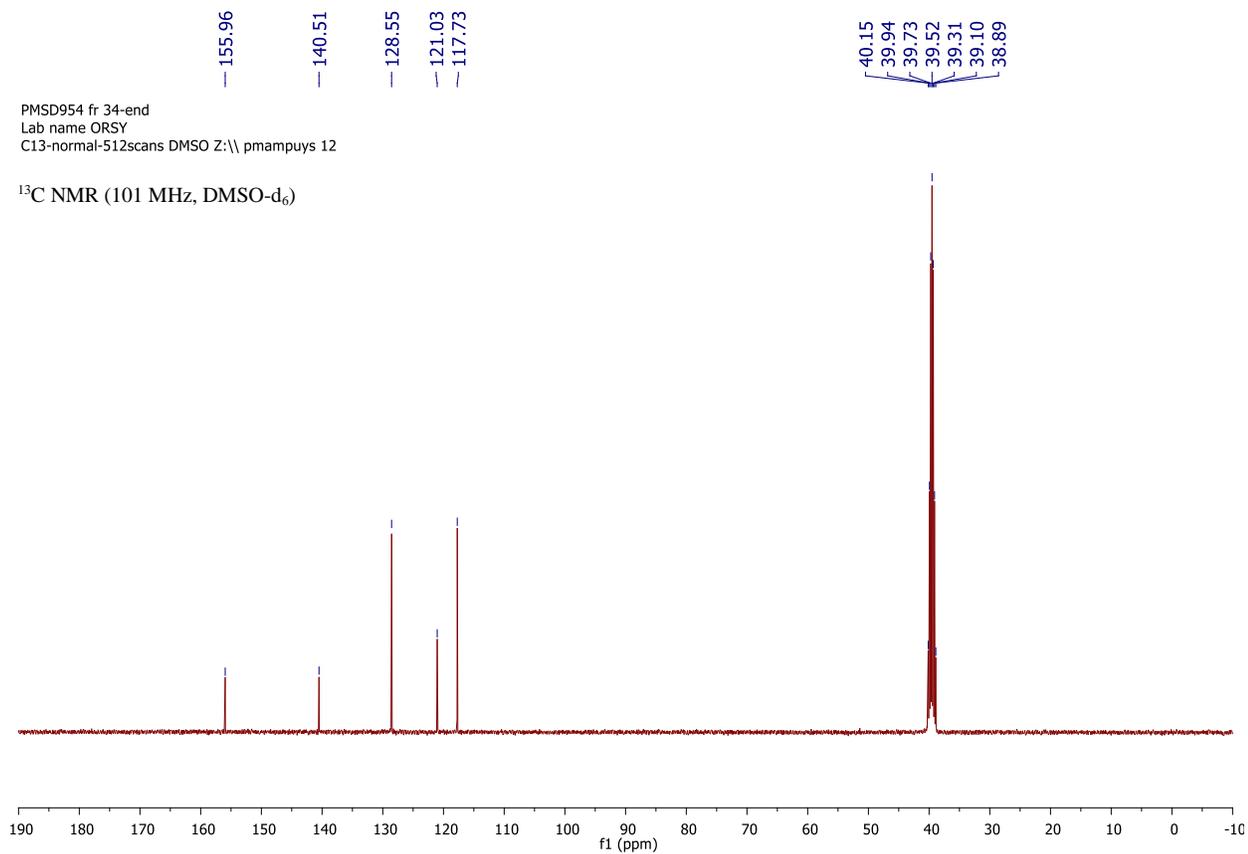
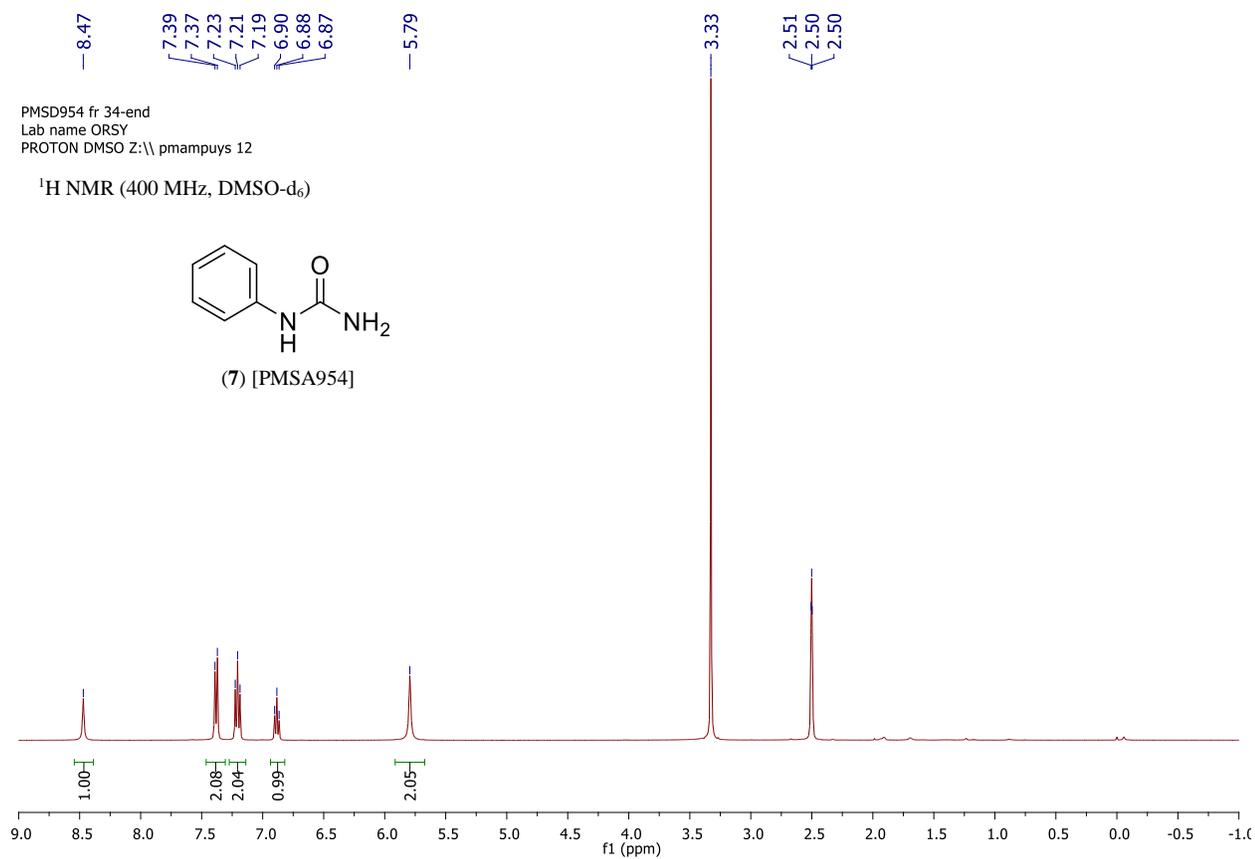


Copies of the NMR spectra





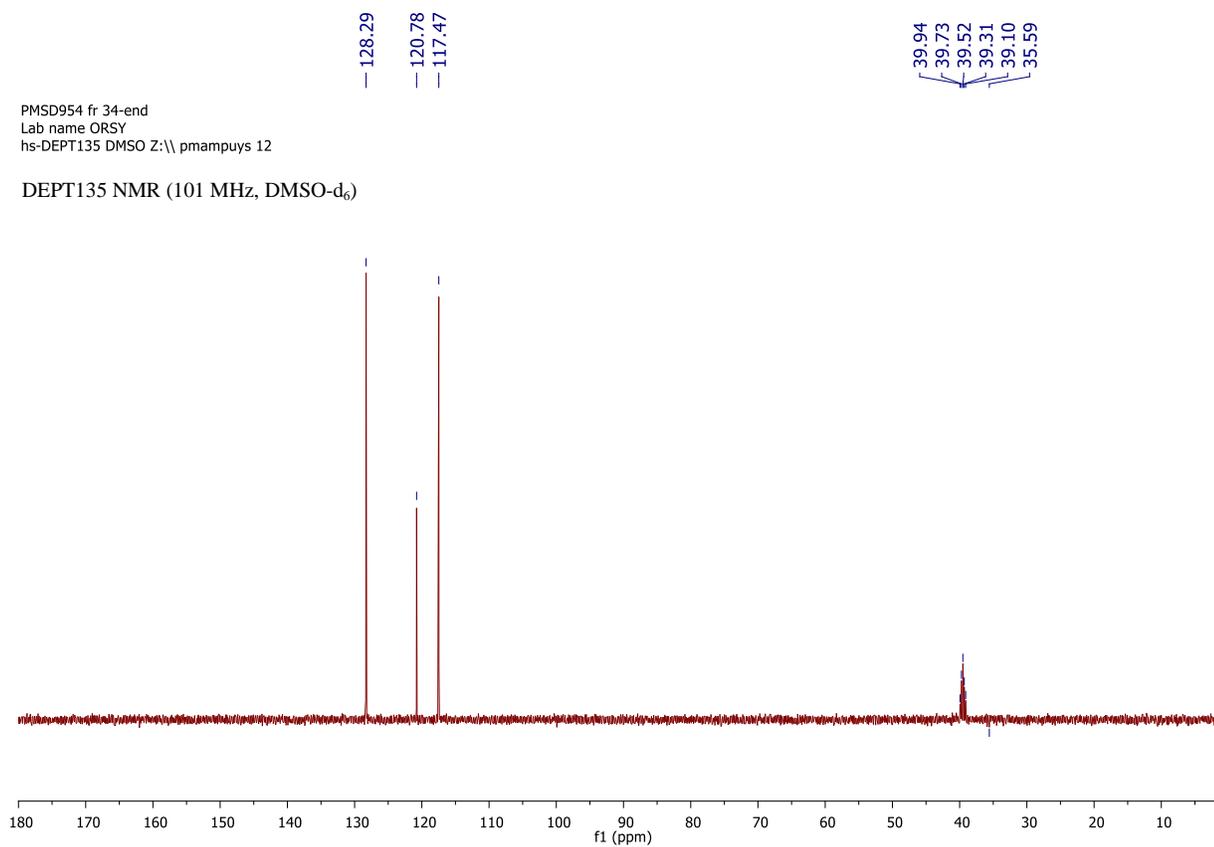
Copies of the NMR spectra



Copies of the NMR spectra

PMSD954 fr 34-end
Lab name ORSY
hs-DEPT135 DMSO Z:\\ pmampuy 12

DEPT135 NMR (101 MHz, DMSO-d₆)



COSY NMR (400 MHz, DMSO-d₆)

