

Supporting Information

Organo-Redox-Catalysis for the difunctionalization of alkenes and oxidative Ritter reactions by C-H functionalization

Sensheng Liu, and Martin Klussmann*

Max-Planck-Institut für Kohlenforschung, 45470 Mülheim/Ruhr, Germany

E-mail: klusi@mpi-muelheim.mpg.de

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Supplementary Methods

General Information

Unless otherwise indicated, all reagents and solvents were purchased from commercial distributors and used as received. Solvents (hexanes, ethyl acetate) used for column chromatography were of technical grade and used after distillation in a rotary evaporator.

TLC was used to check the reactions for full conversion and was performed on Macherey-Nagel Polygram Sil G/UV254 thin layer plates. TLC spots were visualized by UV-light irradiation or used of Phosphomolybdic acid hydrate after heated.

Flash column chromatography was carried out using Merck Silica Gel 60 (40-63 μm). Yields refer to pure isolated compounds.

^1H and ^{13}C NMR spectra were measured with Bruker AV 300 spectrometer, Bruker AV 500 spectrometer, Bruker AV 600 spectrometer. All chemical shifts are given in ppm downfield relative to TMS and were referenced to the solvent residual peaks.^[1] ^1H NMR chemical shifts are designated using the following abbreviations as well as their combinations: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. For ^{13}C NMR data the following abbreviations are used: p = primary (CH_3), s = secondary (CH_2), t = tertiary (CH), q = quaternary (C).

High resolution mass spectra were recorded with a Bruker APEX III FTICR-MS or a Finnigan SSQ 7000 quadrupole MS or a Finnigan MAT 95 double focusing sector field MS instrument.

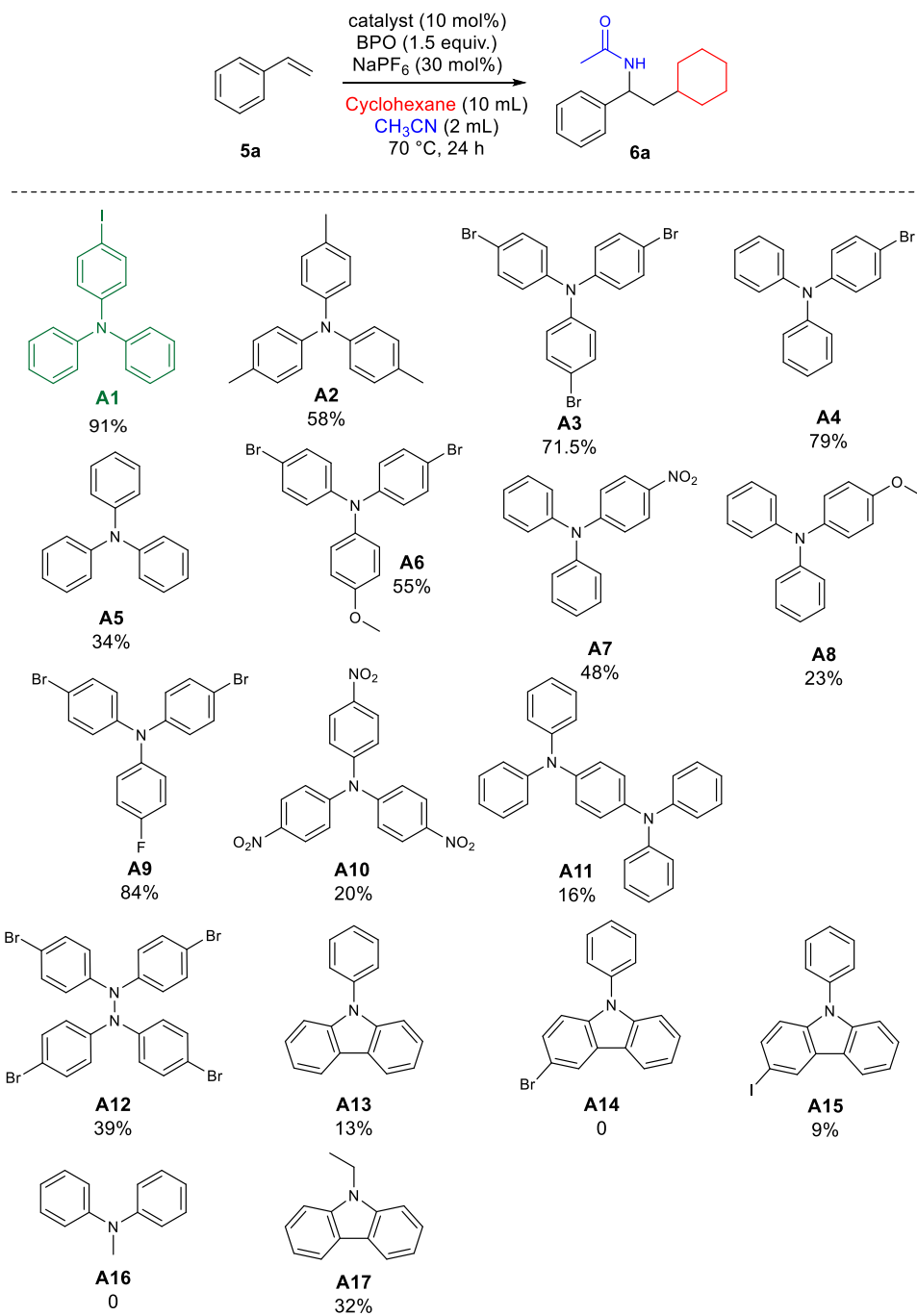
Cyclic Voltammetry (CV) were measured with the BAS Epsilon potentiostat.

Benzoyl peroxide (BPO, 75% in H_2O) and Sodium hexafluorophosphate (NaPF_6) was purchased from Sigma-Aldrich and used directly without further purification.

Tris(4-bromophenyl)ammoniumyl hexachloroantimonate ($\text{TBPA}^+\cdot$) was purchased from Sigma-Aldrich and used directly without further purification.

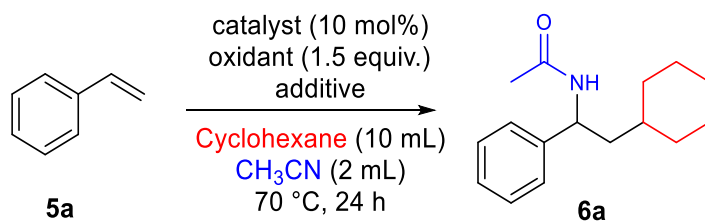
Optimization of reaction conditions

Table S1: Optimization of amine catalysts^[a]



[a] 1a (0.5 mmol, 57 μ L), BPO (0.75 mmol, 1.5 equiv.), catalyst (10 mol%), NaPF₆ (0.15 mmol), cyclohexane (10 mL), CH₃CN (2 mL), 70 °C, 24 h. Yields were determined by ¹H NMR spectroscopic analysis of the crude reaction mixture relative to internal standard 1,3,5-Trimethoxybenzene.

Table S2: Optimization of other reaction conditions



| Entry | Catalyst | Oxidant | Additive | Yield (%) |
|-------------------|---------------|---------|---|-----------------------|
| 1 | A1 | BPO | NaOTf (30 mol%) | 8.4 |
| 2 | A1 | BPO | NaBF ₄ (30 mol%) | 47 |
| 3 | A1 | BPO | ^t Bu ₄ NPF ₆ (30 mol%) | 0 |
| 4 | A1 | BPO | NaOBz (30 mol%) | 0 |
| 5 | A1 | BPO | NaSbF ₆ (30 mol%) | 16 |
| 6 | A1 | BPO | NaPF ₆ (30 mol%) | 91(87) ^[b] |
| 7 | A1 | BPO | NaPF ₆ (50 mol%) | 88 |
| 8 | A1 | BPO | NaPF ₆ (10 mol%) | 63 |
| 9 | A1 | BPO | NaPF ₆ (5 mol%) | 50 |
| 10 | A1 (5 mol%) | BPO | NaPF ₆ (30 mol%) | 66 |
| 11 | A1 (2.5 mol%) | BPO | NaPF ₆ (30 mol%) | 26 |
| 12 | A1 (1.0 mol%) | BPO | NaPF ₆ (30 mol%) | 23 |
| 13 | A1 (0.5 mol%) | BPO | NaPF ₆ (30 mol%) | 8 |
| 14 ^[c] | A1 | BPO | NaPF ₆ (30 mol%) | 89 |
| 15 ^[d] | A1 | BPO | NaPF ₆ (30 mol%) | 2 |
| 16 | A1 | TBHP | NaPF ₆ (30 mol%) | 0 |
| 17 | A1 | TBPP | NaPF ₆ (30 mol%) | <10 |
| 18 | A1 | DTBP | NaPF ₆ (30 mol%) | <10 |
| 19 ^[e] | A1 | DTBP | NaPF ₆ (30 mol%) | 32 |
| 20 | - | BPO | NaPF ₆ (30 mol%) | 0 |
| 21 | A1 | - | NaPF ₆ (30 mol%) | 0 |
| 22 | A1 | BPO | - | 0 |

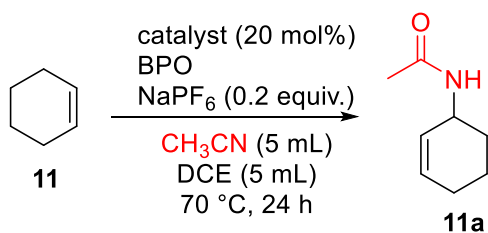
[a] 1a (0.5 mmol, 57 μ L), BPO (0.75 mmol, 1.5 equiv.), catalyst (10 mol%), additive, cyclohexane (10 mL), CH₃CN (2 mL), 70 °C, 24 h. Yields were determined by ¹H NMR spectroscopic analysis of the crude reaction mixture relative to internal standard 1,3,5-Trimethoxybenzene. [b] Isolated yield. [c] 90 °C. [d] 50 °C. [e] 100 °C.

Additive effect: additive is required (compare entries 1-6) and the NaPF₆ is important for good yields of product 6a. However, others such as NaBF₄, NaSbF₆ and NaOTf give low yield of 6a, NaOBz and ^tBu₄NPF₆ give no desired products. The concentration of additive seems to be less important, compare entries 6-9.

Catalyst loading: The lower concentration of catalyst, the lower yields were got.

Oxidants: BPO is the best oxidant in this reaction.

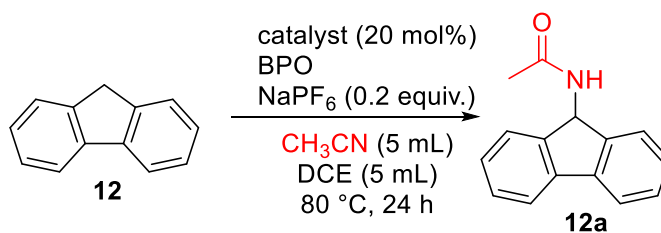
Table S3: Optimization of allylic C-H Ritter reaction



| Entry | Catalyst | Oxidant | Solvent | Yield (%) |
|------------------|-----------|------------------|--|-----------|
| 1 | A1 | BPO (1.5 equiv.) | CH ₃ CN (10 mL) | 20 |
| 2 | A1 | BPO (2.0 equiv.) | CH ₃ CN (10 mL) | 25 |
| 3 | A2 | BPO (2.0 equiv.) | CH ₃ CN (10 mL) | 36 |
| 4 | A3 | BPO (2.0 equiv.) | CH ₃ CN (10 mL) | 37 |
| 5 | A3 | BPO (2.0 equiv.) | CH ₃ CN (5 mL) + CH ₃ NO ₂ (5 mL) | 0 |
| 6 | A3 | BPO (2.0 equiv.) | CH ₃ CN (5 mL) + THF (5 mL) | 0 |
| 7 | A3 | BPO (2.0 equiv.) | CH ₃ CN (5 mL) + DCE (5 mL) | 71 |
| 8 ^[b] | A3 | BPO (2.0 equiv.) | CH ₃ CN (5 mL) + DCE (5 mL) | 0 |
| 9 | - | BPO (2.0 equiv.) | CH ₃ CN (5 mL) + DCE (5 mL) | 0 |

[a] **11** (0.5 mmol), BPO, catalyst (20 mol%), NaPF₆ (20 mol%), solvent, 70 °C, 24 h. Yields were determined by ¹H NMR spectroscopic analysis of the crude reaction mixture relative to internal standard 1,3,5-Trimethoxybenzene. [b] no NaPF₆.

Table S4: Optimization of benzylic C-H Ritter reaction

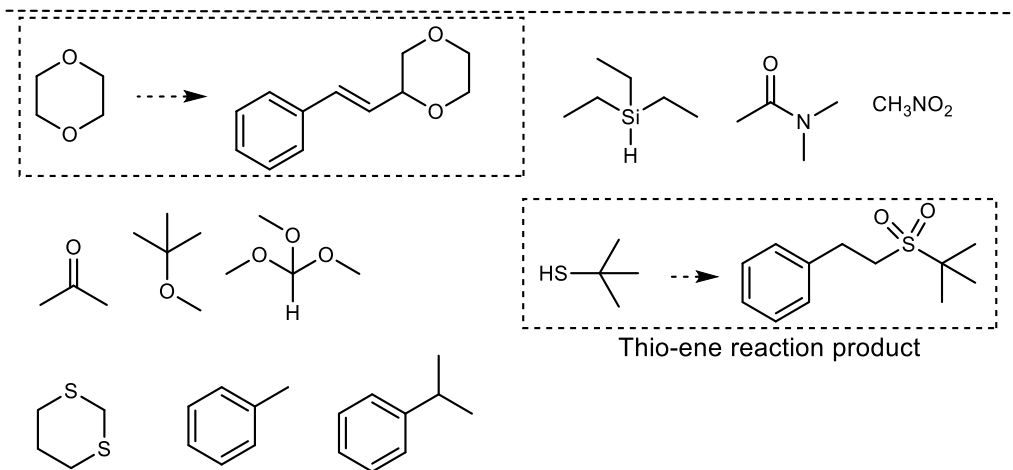
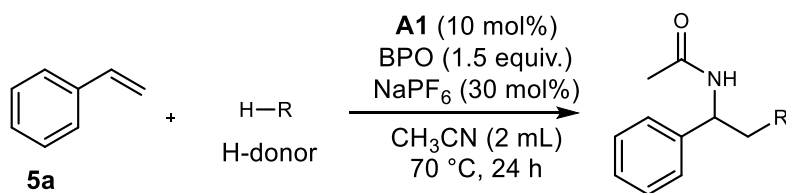


| Entry | Catalyst | Oxidant | Tem/ °C | Yield (%) |
|------------------|-----------|------------------|---------|-----------|
| 1 | A3 | BPO (2.0 equiv.) | 70 | 32 |
| 2 | A3 | BPO (3.0 equiv.) | 70 | 43 |
| 3 | A3 | BPO (3.0 equiv.) | 80 | 70 |
| 4 | A3 | BPO (3.0 equiv.) | 100 | 60 |
| 5 | A1 | BPO (2.0 equiv.) | 80 | 41 |
| 6 | A2 | BPO (2.0 equiv.) | 80 | 15 |
| 7 ^[b] | A3 | BPO (2.0 equiv.) | 80 | 0 |
| 8 | - | BPO (2.0 equiv.) | 80 | 0 |

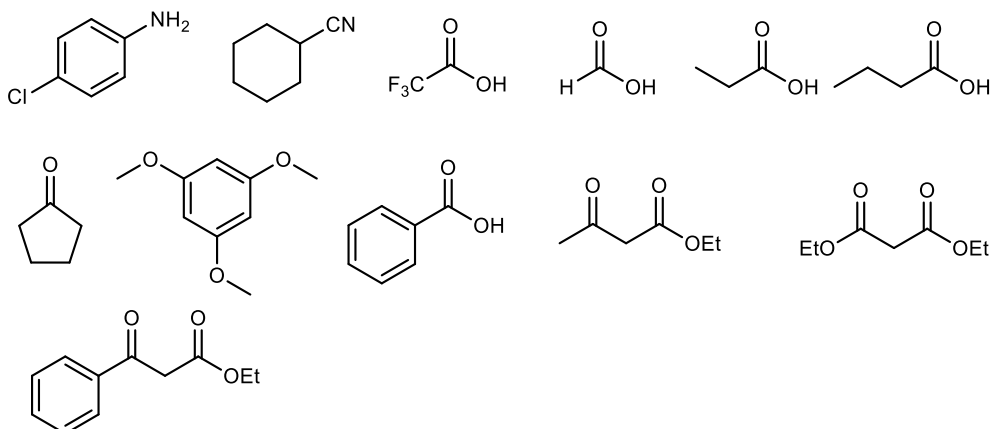
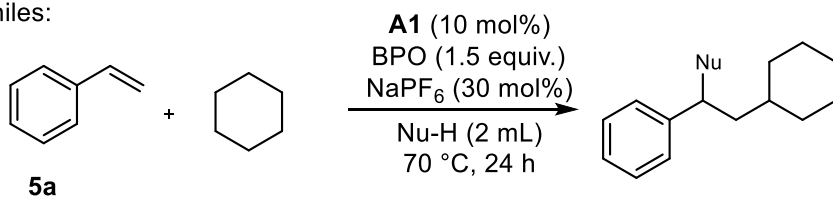
[a] **12** (0.5 mmol), BPO, catalyst (20 mol%), NaPF₆ (20 mol%), DCE (5 mL), CH₃CN (5 mL), 80 °C, 24 h. Yields were determined by ¹H NMR spectroscopic analysis of the crude reaction mixture relative to internal standard 1,3,5-Trimethoxybenzene. [b] no NaPF₆.

Failed examples:

H-donor:

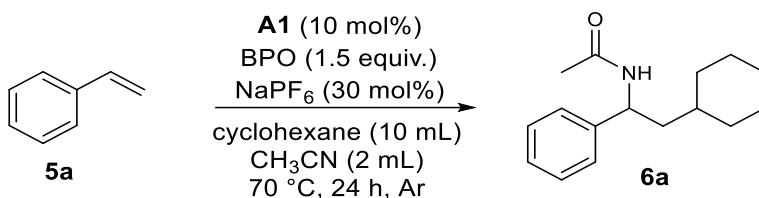


Nucleophiles:



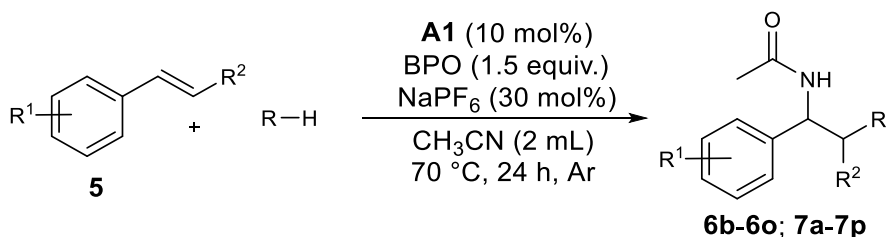
Reaction procedures

Procedure A: synthesis of *N*-(2-cyclohexyl-1-phenylethyl)acetamide.



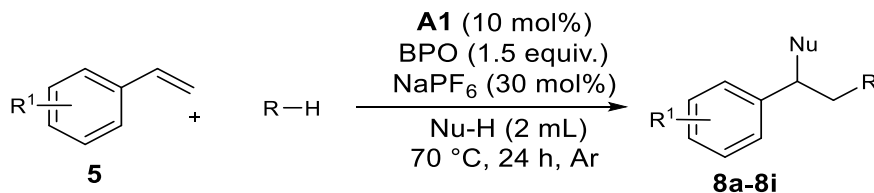
Under argon atmosphere, the styrene **5a** (0.5 mmol, 57 μ L), catalyst **A1** (10 mol%, 0.05 mmol, 18.5 mg), cyclohexane (10 mL), CH₃CN (2 mL), BPO (0.75 mmol, 1.5 equiv.) and NaPF₆ (0.15 mmol, 30 mol%) were added into a 25 mL glass tube. The reaction mixture was stirred at 70 °C for 24 h under Ar atmospheres. After the reaction was fully completed, the mixture was cooled to room temperature and concentrated under reduced pressure to give a crude product. The residue was further purified by silica gel column with *iso*-hexane/ethyl acetate (from 10:1 to 1:1) to give the desired products **6a**.

General procedure B: synthesis of *N*-(1-phenylalkyl)acetamides.



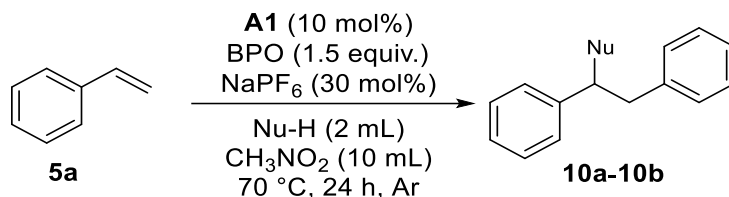
Under argon atmosphere, the styrenes **5** (0.5 mmol), catalyst **A1** (10 mol%, 0.05 mmol, 18.5 mg), R-H (10 mL), CH₃CN (2 mL), BPO (0.75 mmol, 1.5 equiv.) and NaPF₆ (0.15 mmol, 30 mol%) were added into a 25 mL glass tube. The reaction mixture was stirred at 70 °C for 24 h under Ar atmospheres. After the reaction was fully completed, the mixture was cooled to room temperature and concentrated under reduced pressure to give a crude product. The residue was further purified by silica gel column with *iso*-hexane/ethyl acetate (from 10:1 to 1:1) to give the desired products **6b-6o** and **7a-7p**.

General procedure C: synthesis of radicals and nucleophiles addition.



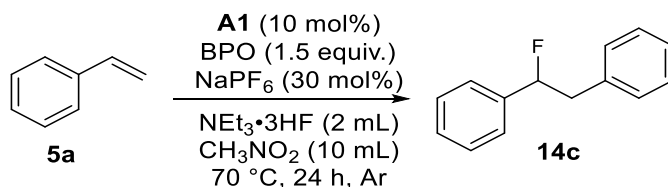
Under argon atmosphere, the styrenes **5** (0.5 mmol), catalyst **A1** (10 mol%, 0.05 mmol, 18.5 mg), nucleophiles (2 mL), R-H (10 mL), BPO (0.75 mmol, 1.5 equiv.) and NaPF₆ (0.15 mmol, 30 mol%) were added into a 25 mL glass tube. The reaction mixture was stirred at 70 °C for 24 h under Ar atmospheres. After the reaction was fully completed, the mixture was cooled to room temperature and concentrated under reduced pressure to give a crude product. The residue was further purified by silica gel column with *iso*-hexane/ethyl acetate (from 50:1 to 10:1) to give the desired products **8a-8i**.

General procedure D: synthesis of phenyl radicals and nucleophiles addition.



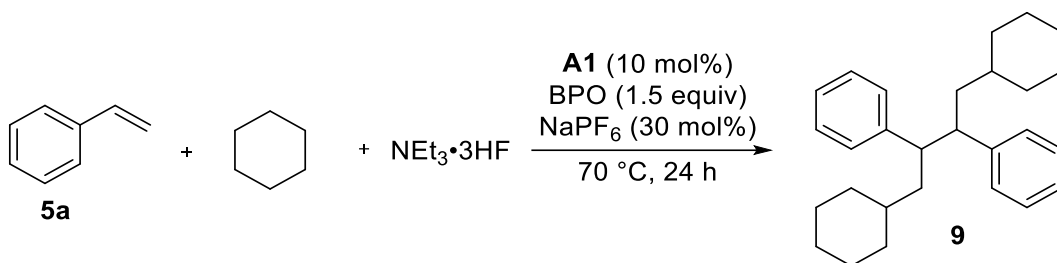
Under argon atmosphere, the styrene **5a** (0.5 mmol, 57 μL), catalyst **A1** (10 mol%, 0.05 mmol, 18.5 mg), nucleophiles (2 mL), CH₃NO₂ (10 mL), BPO (0.75 mmol, 1.5 equiv.) and NaPF₆ (0.15 mmol, 30 mol%) were added into a 25 mL glass tube. The reaction mixture was stirred at 70 °C for 24 h under Ar atmospheres. After the reaction was fully completed, the mixture was cooled to room temperature and concentrated under reduced pressure to give a crude product. The residue was further purified by silica gel column with *iso*-hexane/ethyl acetate (from 50:1 to 10:1) to give the desired products **10a** and **10b**.

Synthesis of (1-fluoroethane-1,2-diyl)dibenzene.



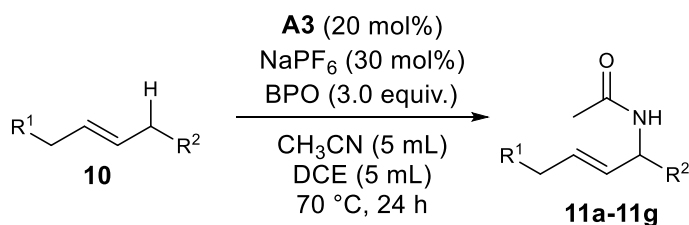
Under argon atmosphere, the styrene **5a** (0.5 mmol, 57 μL), catalyst **A1** (10 mol%, 0.05 mmol, 18.5 mg), NEt₃·3HF (2 mL), CH₃NO₂ (10 mL), BPO (0.75 mmol, 1.5 equiv.) and NaPF₆ (0.15 mmol, 30 mol%) were added into a 25 mL glass tube. The reaction mixture was stirred at 70 °C for 24 h under Ar atmospheres. After the reaction was fully completed, the mixture was cooled to room temperature and concentrated under reduced pressure to give a crude product. The residue was further purified by silica gel column with *iso*-hexane/ethyl acetate (from 500:1 to 100:1) to give the desired product **14c** in the yield of 26 %.

Synthesis of (1,4-dicyclohexylbutane-2,3-diyl)dibenzene.



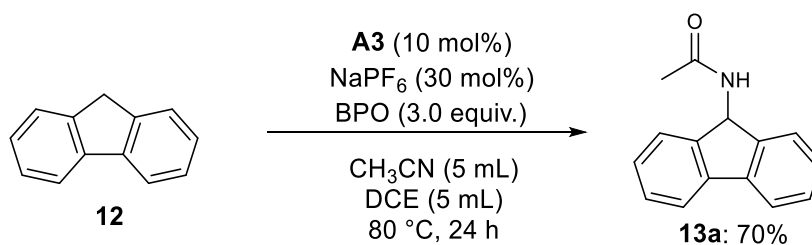
Under argon atmosphere, the styrene **5a** (0.5 mmol), catalyst **A1** (10 mol%, 0.05 mmol, 18.5 mg), $\text{NEt}_3\cdot 3\text{HF}$ (2 mL), cyclohexane (10 mL), BPO (0.75 mmol, 1.5 equiv.) and NaPF_6 (0.15 mmol, 30 mol%) were added into a 25 mL glass tube. The reaction mixture was stirred at 70 °C for 24 h under Ar atmospheres. After the reaction was fully completed, the mixture was cooled to room temperature and concentrated under reduced pressure to give a crude product. The residue was further purified by silica gel column with *iso*-hexane/ethyl acetate (from 500:1 to 100:1), not giving the desired fluoride but the dimer **9** in the yield of 31%.

General procedure E: oxidative Ritter reaction of allylic C-H bond.



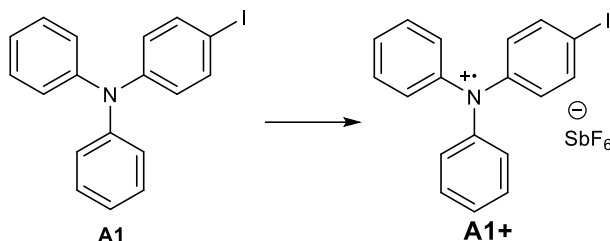
Under argon atmosphere, the olefins **10** (0.5 mmol), catalyst **A3** (20 mol%, 0.05 mmol, 24.1 mg), CH_3CN (5 mL), DCE (5 mL), BPO (1.5 mmol, 3.0 equiv.) and NaPF_6 (0.15 mmol, 30 mol%) were added into a 25 mL glass tube. The reaction mixture was stirred at 70 °C for 24 h under Ar atmospheres. After the reaction was fully completed, the mixture was cooled to room temperature and concentrated under reduced pressure to give a crude product. The residue was further purified by silica gel column with *iso*-hexane/ethyl acetate (from 50:1 to 10:1) to give the desired products **11a-11g**. By using (*Z*)-decene as starting material, (*E*)-**11d** and (*E*)-**11c** were isolated.

Oxidative Ritter reaction of *N*-(9*H*-fluoren-9-yl)acetamide.



Under argon atmosphere, the 9*H*-fluorene **12** (0.5 mmol, 83 mg), catalyst **A3** (20 mol%, 0.05 mmol, 24.1 mg), CH₃CN (5 mL), DCE (5 mL), BPO (1.5 mmol, 3.0 equiv.) and NaPF₆ (0.15 mmol, 30 mol%) were added into a 25 mL glass tube. The reaction mixture was stirred at 80 °C for 24 h under Ar atmospheres. After the reaction was fully completed, the mixture was cooled to room temperature and concentrated under reduced pressure to give a crude product. The residue was further purified by silica gel column with *iso*-hexane/ethyl acetate (from 50:1 to 10:1) to give the desired product **13a**.

Synthesis of triarylamine radical cation salt **A1+** ^[2]

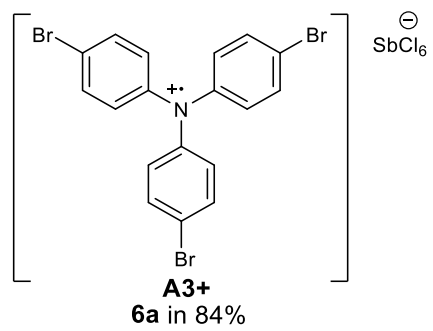
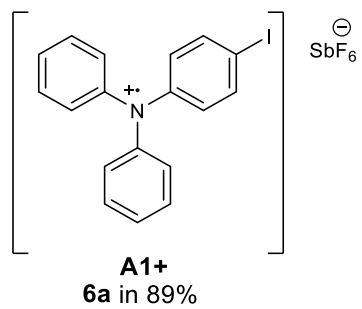


A1+. To a solution of **A1** (185 mg, 0.5 mmol, 1.0 equiv.) in dry CH₂Cl₂ (5 mL) under Ar atmosphere was added AgSbF₆ (180.4 mg, 0.525 mmol, 1.05 equiv.) at room temperature. The slightly yellowish reaction mixture immediately turned dark blue and the reaction was stirred for about one minute. After removal of insoluble contents by filtration, product **A1+** was obtained by precipitation from CH₂Cl₂/hexanes as a deep blue powder (275.7 mg, 91%). HRMS (ESI, DCM/CH₃CN, positive mode) *m/z* for C₁₈H₁₄NI^{•+}(M⁺) calcd. 371.0165, found 371.0163; for SbF₆⁻(M⁻) calcd. 234.8949, found 234.8949. In the ¹H NMR, only a very broad signal around 8.5 ppm -6.5 ppm could be observed, and no signal in the ¹³C NMR. No signals from the starting material (**A1**) could be seen, suggesting that the product is the pure ammoniumyl radical cation, free of the closed-shell starting material.

Synthesis of *N*-(3-cyano-1-phenylpropyl)acetamide catalyzed by triarylamine radical cation salts.



Under argon atmosphere, the styrene **5a** (0.5 mmol, 57 μL), catalyst (10 mol%, 0.05 mmol), CH₃CN (12 mL), BPO (0.75 mmol, 1.5 equiv.), NaPF₆ (0.15 mmol, 30 mol%) were added into a 25 mL glass tube. The reaction mixture was stirred at 70 °C for 24 h under Ar atmospheres. After the reaction was fully completed, the mixture was cooled to room temperature and concentrated under reduced pressure to give a crude product. 1,3,5-Trimethoxybenzene (10 mg) added as internal standard for NMR, yields base on the NMR yield.



Cyclic voltammograms

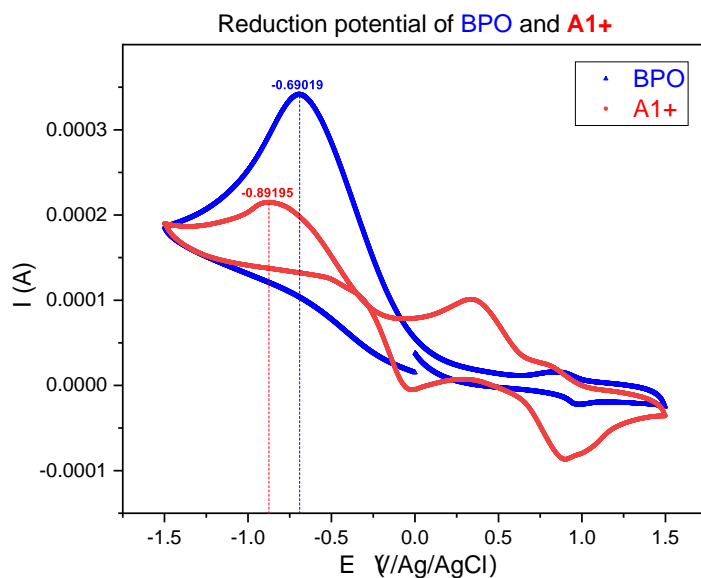


Figure 1. Cyclic voltammograms shows the reduction potential of A1+ and BPO. Two platinized Pt and Carbon wires as a counter and working electrode with a Ag/AgCl electrode as a reference were used. The cyclic voltammetry (CV) was conducted from -1.5 V to 1.5 V with a scan rate of 200 mV/s⁻¹. A1+ (0.03 mmol) or BPO (0.03 mmol) in 3 mL in CH₃CN of LiClO₄ (0.1 M) under Ar.

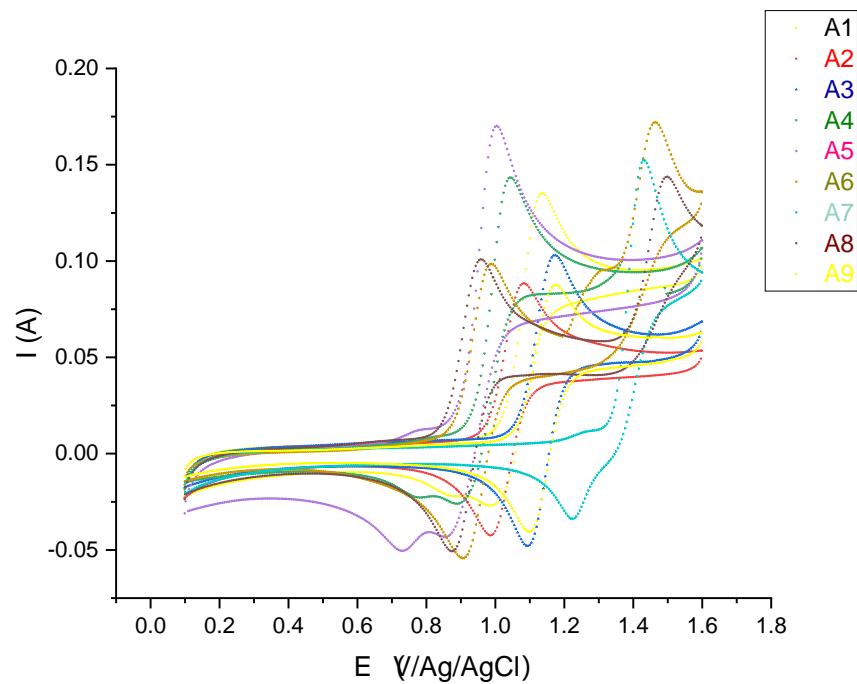
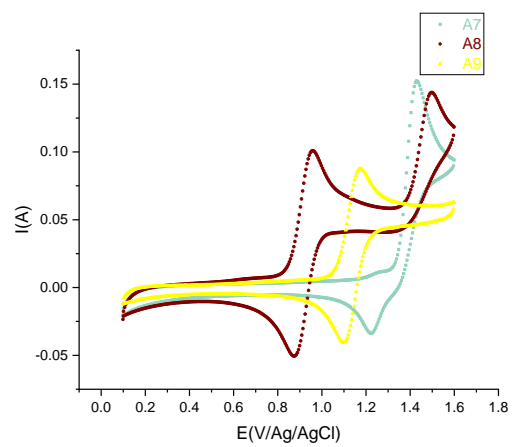
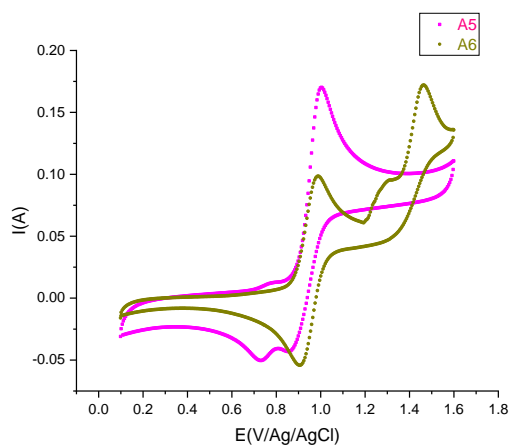
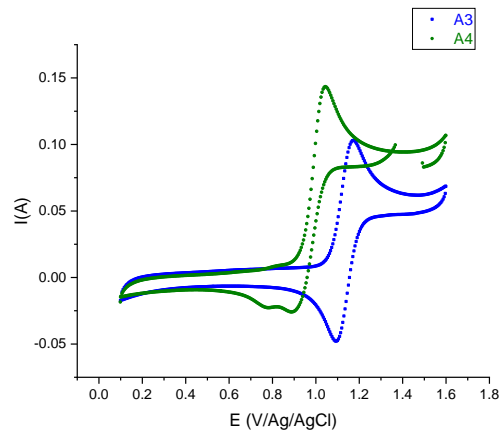
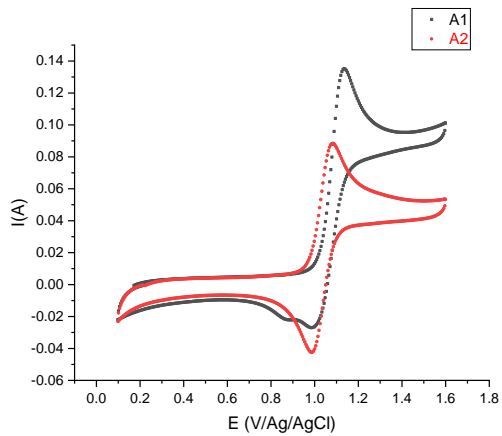


Figure 2. Cyclic voltammogram showing the structure-function relationship on the reduction and oxidation potential of amine catalysts. Two platinized Pt wires as a counter and working electrode with a Ag/AgCl electrode as a reference were used. The cyclic voltammetry (CV) was conducted from 0 V to 1.5 V with a scan rate of 100 mVs⁻¹. Triarylamine (0.2 mmol), tetrabutylammonium hexafluorophosphate (0.1 M) in CH₃CN under Ar.



Mechanistic study

NMR study of BPO and catalyst in CD₃CN at 70 °C.

In an NMR tube, BPO (0.1 mmol) was added to CD₃CN (0.5 mL) and a first ¹H-NMR spectrum was measured as a control. The NMR tube was then heated at 70 °C in an oil-bath and ¹H-NMR spectra were measured after 2 h and 10 h, respectively, each time by removing the NMR tube from the oil-bath and measuring the spectra at ambient temperature. See the red line in the scheme below.

NMR studies of BPO decompose in CD₃CN

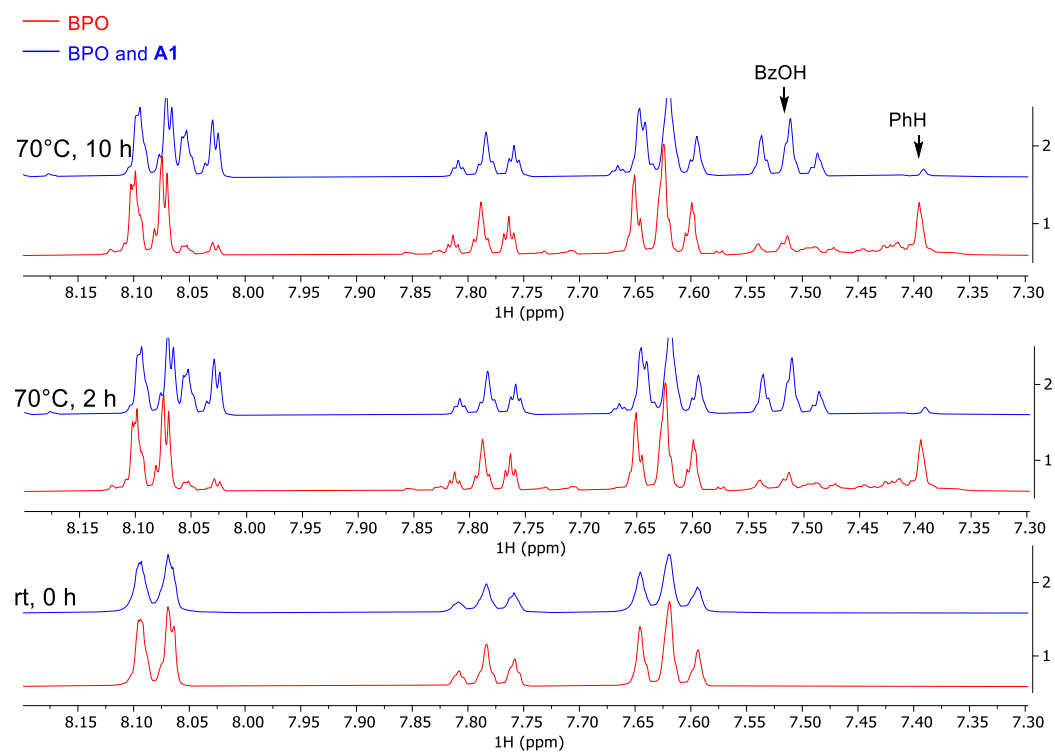


Table S5: Relative amounts of BPO, BzOH, and PhH for each experiment after 2 h and 10 h

A: BPO only; B: BPO + A1.

| Reaction | BPO (mmol) | | | | | BzOH (mmol) | | | | | PhH (mmol) | | | | |
|----------|------------|-------|-------|-------|-------|-------------|--------|-------|-------|-------|------------|-------|-------|-------|-------|
| | 0 h | 0.5 h | 1 h | 2 h | 10 h | 0 h | 0.5 h | 1 h | 2 h | 10 h | 0 h | 0.5 h | 1h | 2 h | 10 h |
| A | 0.1 | 0.097 | 0.087 | 0.073 | 0.039 | 0 | 0.005 | 0.017 | 0.025 | 0.081 | 0 | 0.003 | 0.021 | 0.023 | 0.076 |
| B | 0.1 | 0.088 | 0.079 | 0.063 | 0.041 | 0 | 0.0027 | 0.05 | 0.079 | 0.132 | 0 | 0.001 | 0.003 | 0.01 | 0.012 |

BPO (0.1 mmol), catalyst (10 mol%) in 0.5 mL CD₃CN.

Amount of BPO, BzOH, and PhH.

NMR yields based on the integration of BPO.

Table S6: % conversion of BPO, BzOH, and PhH for each experiment after 2 h and 10 h

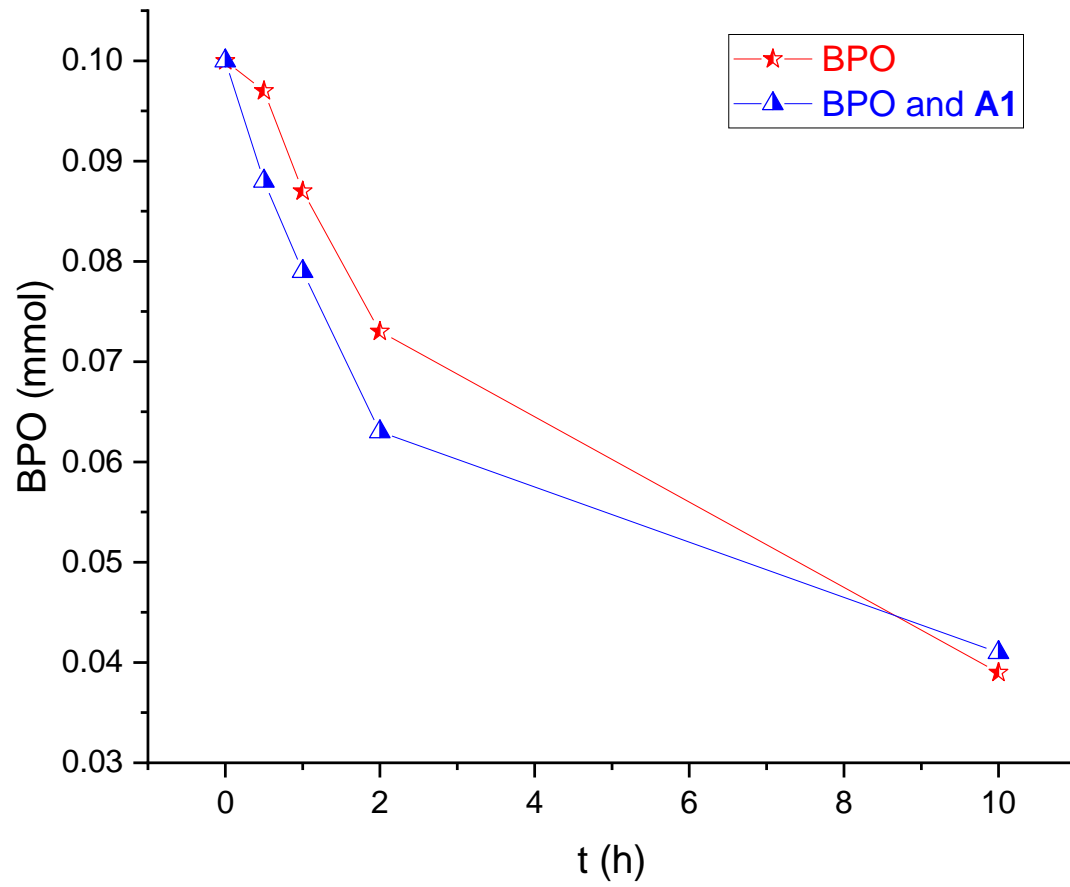
A: BPO only; B: BPO + A1.

| Reaction | BPO | | | | | BzOH | | | | | PhH | | | | |
|----------|-----|-------|-----|-----|------|------|-------|------|-------|-------|-----|-------|-------|-------|------|
| | 0 h | 0.5 h | 1 h | 2 h | 10 h | 0 h | 0.5 h | 1 h | 2 h | 10 h | 0 h | 0.5 h | 1h | 2 h | 10 h |
| A | 1 | 3% | 13% | 27% | 60% | 0 | 2.5% | 8.5% | 12.5% | 40.5% | 0 | 1.5% | 10.5% | 11.5% | 38% |
| B | 1 | 12% | 21% | 37% | 59% | 0 | 1.35% | 25% | 39.5% | 66% | 0 | 0.5% | 1.5% | 5% | 6% |

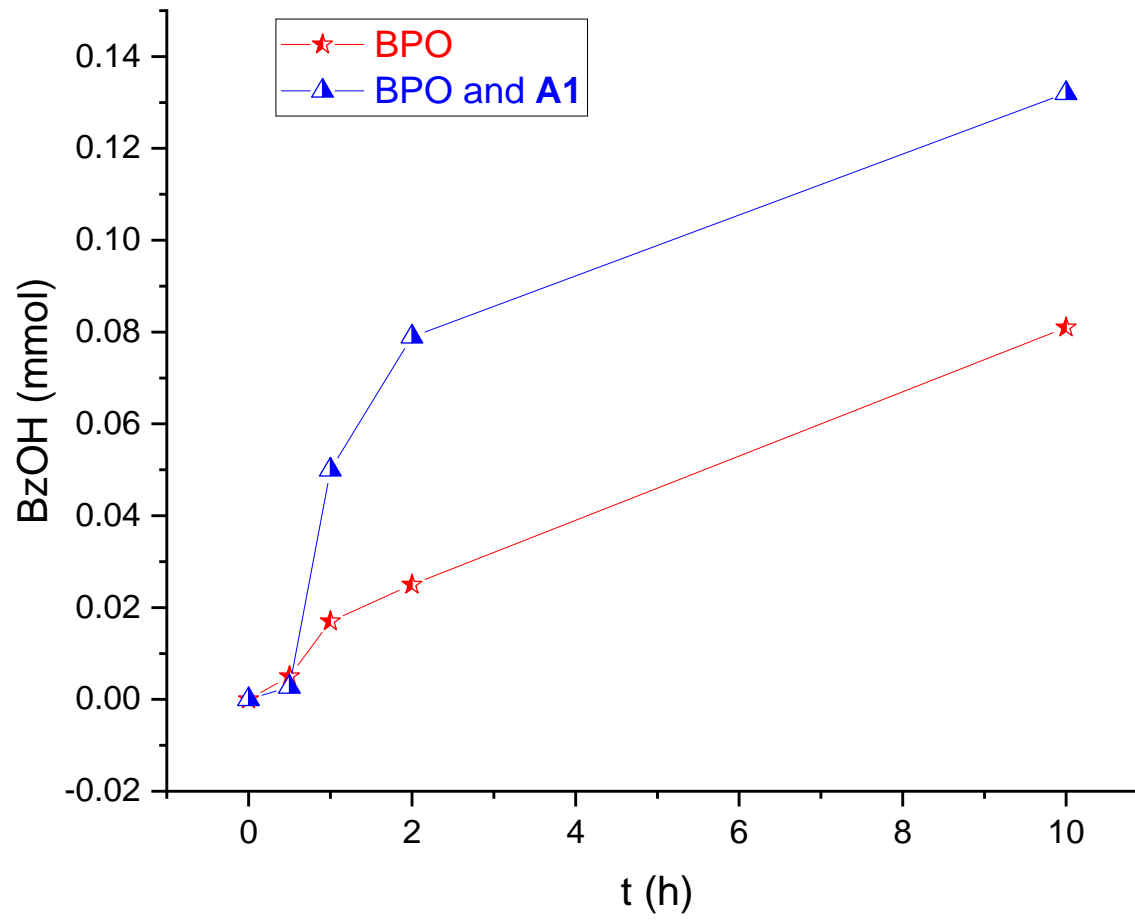
BPO (0.1 mmol), catalyst (10 mol%) in 0.5 mL CD₃CN.

Amount of BPO, BzOH, and PhH.

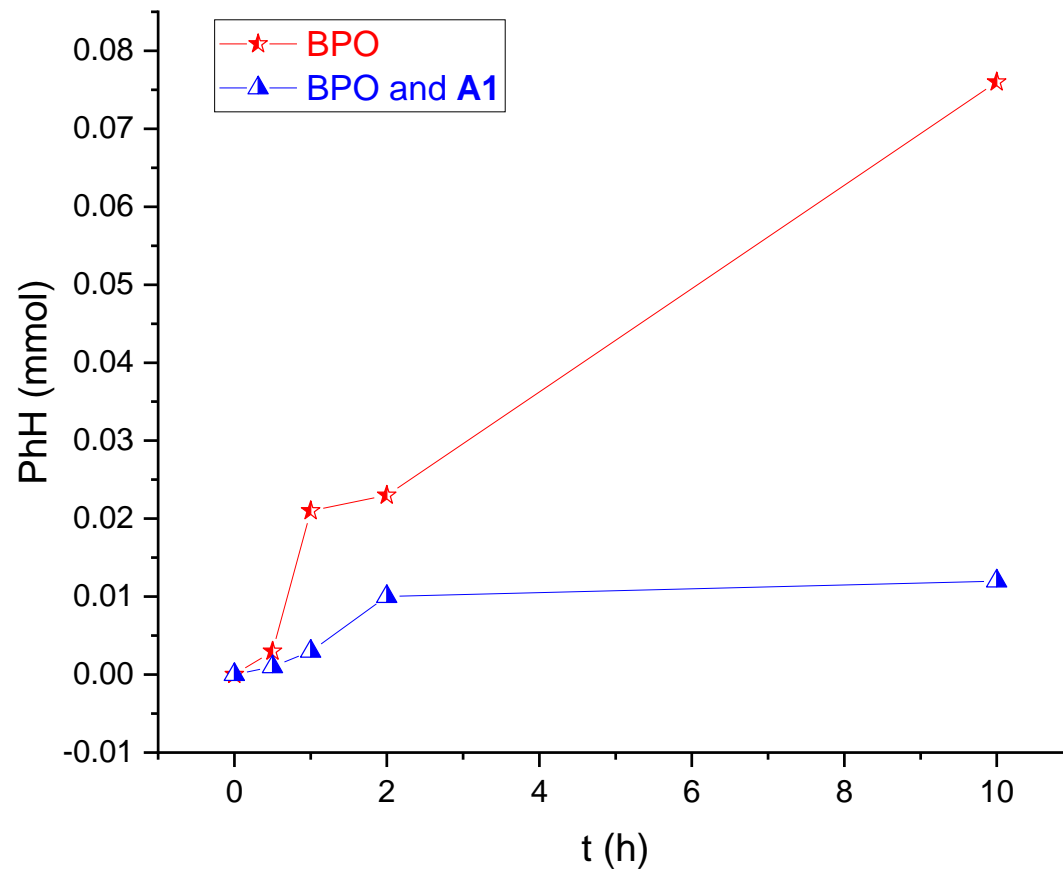
% conversion based on the integration of BPO.



Amount of BPO in different time

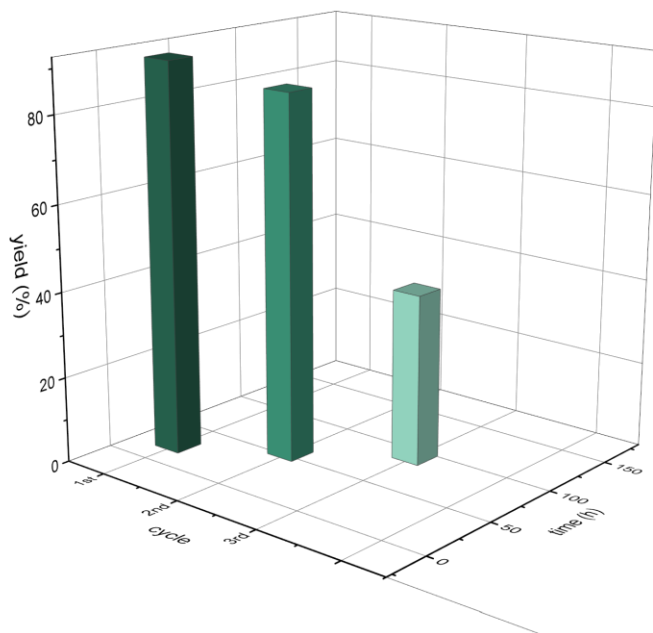


Amount of BzOH in different time

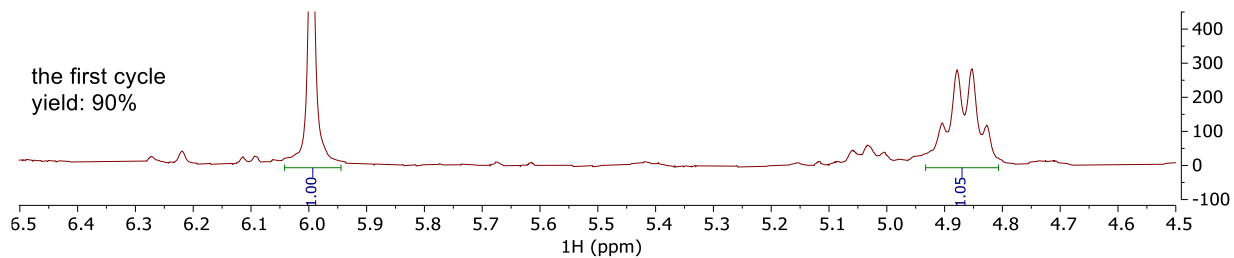
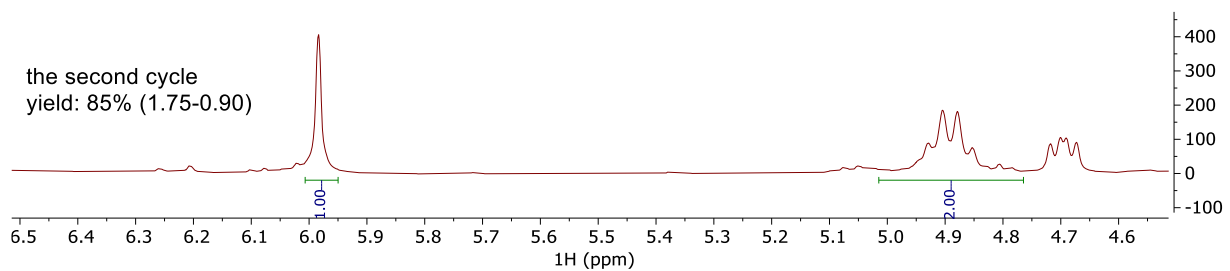
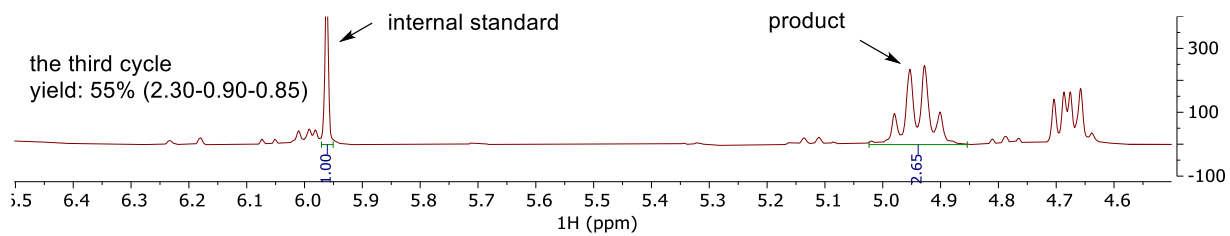


Amount of PhH in different time

Details of the repetitive cycles experiment



- The 1st cycle: **1a** (0.5 mmol), **A1** (10 mol%), BPO (1.5 equiv.), NaPF₆ (30 mo%), cyclohexane (10 mL), CH₃CN (2 mL), 70 °C, 24 h. **Yield: 90%**. Determined by ¹H NMR spectroscopic analysis of the crude reaction mixture relative to internal standard 1,3,5-trimethoxybenzene.
- The 2nd cycle: **1a** (0.5 mmol), **A1** (10 mol%), BPO (1.5 equiv.), NaPF₆ (30 mo%), cyclohexane (10 mL), CH₃CN (2 mL), 70 °C, after 24 h, **1a** (0.5 mmol) and BPO (1.5 equiv.) were added again, keep the reaction at 70 °C for another 30 h. **Yield: 85%**. Determined by ¹H NMR spectroscopic analysis of the crude reaction mixture relative to internal standard 1,3,5-trimethoxybenzene.
- The 3rd cycle: **1a** (0.5 mmol), **A1** (10 mol%), BPO (1.5 equiv.), NaPF₆ (30 mo%), cyclohexane (10 mL), CH₃CN (2 mL), 70 °C, after 24 h, **1a** (0.5 mmol) and BPO (1.5 equiv.) were added again, after 30 h at 70 °C, added **1a** (0.5 mmol) and BPO (1.5 equiv.) again, keep the reaction at 70 °C for another 38 h. **Yield: 55%**. Determined by ¹H NMR spectroscopic analysis of the crude reaction mixture relative to internal standard 1,3,5-trimethoxybenzene.

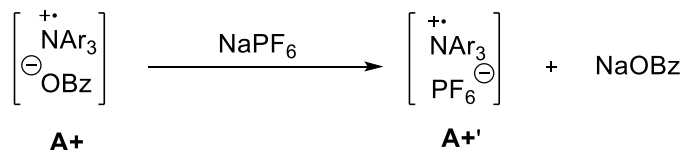


¹H NMR yield for repetitive cycle experiments

Anions exchange between PF_6^- and OBz^-

White particulate can be isolated after the reaction cooled down. Washed the white particulate three times with acetonitrile, acetone, and dichloromethane. Analyzing it by HRMS. From MS and HRMS spectral, only OBz^- can be detected. Which means a little amount of NaOBz was formed. Besides, NaOBz is insoluble but NaPF_6 is soluble in cyclohexane and acetonitrile.

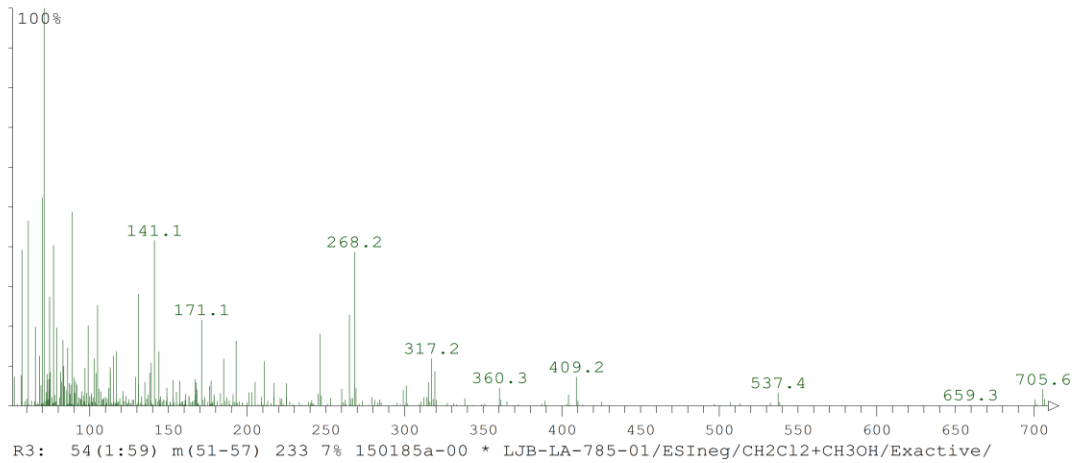
Triarylamine catalysts react with BPO to form intermediate radical cation \mathbf{A}^+ , which undergoes anion exchange to give more stable radical cation \mathbf{A}'^+ and NaOBz . This is the reason why the additive NaPF_6 is indispensable in this reaction system.



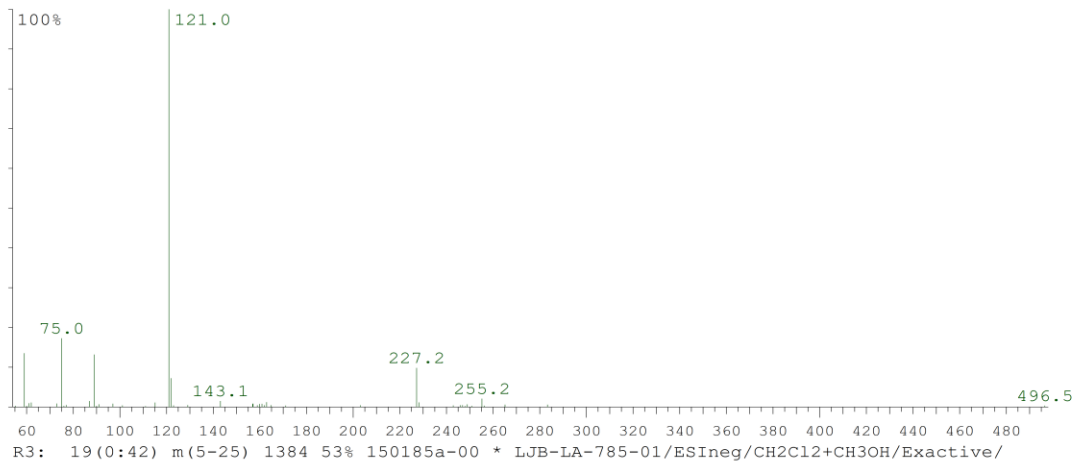
NaOBz

electrospray ionisation pos. ions
 Characteristic Ions:
 57, 61, 71, 89, 141, 171, 268, 409,
 537 et. al. possible
 m/z 23= [Na] - out of measurement range

Datum: 29.10.2020
 Analyse: 150185a-00
 Sigel: LJB-LA-785-01
 KMA: Liu, Sensheng
 Methode: ESI pos+neg
 Lösungsmittel: CH3OH+CH2Cl2
 Spektrometer: Exactive
 Auswerter: Marcus, Tel:2243



electrospray ionization neg. ions
 Characteristic Ions:
 59
 75
 89
 121 = [C7H5O2]- = [144 - Na] possible
 227
 255 et. al. possible
 Other measurements will follow (HRMS)



Mass to be matched (m/z): 121.029550 Charge: -1

Mass Tolerance: ± 0.005550
Restriction of atom numbers:
C H O
1-100 1-100 1-10
Number of calculated Formulas: 1

| Formula | Diff. (ppm) | theor. m/z |
|----------|-------------|------------|
| C7 H5 O2 | -0.37 | 121.029505 |

Datum: 29.10.2020
Analyse: 150185b-00

Sigel: LJB-LA-785-01
KMA: Liu, Sensheng

Method: HR-MS
Ionis. : ESIneg
solvent : CH2Cl2 + CH3OH
Spectrometer: Exactive

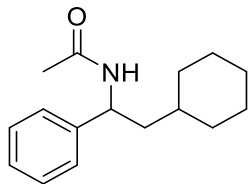
Auswerter: Marcus, Tel:2243

--- possible element composition---

suggestion:
121 = [C7H5O2]-

Characterization Data

N-(2-cyclohexyl-1-phenylethyl)acetamide (**6a**, CAS: 2400221-26-1)



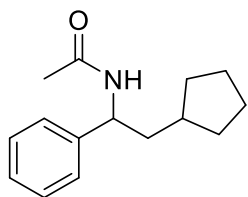
Following the **general procedure A**, white solid (105.5 mg, 86%)

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.27 (d, *J* = 8.6 Hz, 1H), 7.39–7.30 (m, 4H), 7.28–7.22 (m, 1H), 4.92 (td, *J* = 9.2, 5.8 Hz, 1H), 1.88 (s, 3H), 1.84–1.59 (m, 6H), 1.50 (ddd, *J* = 13.8, 8.1, 5.8 Hz, 1H), 1.35–1.13 (m, 4H), 0.98–0.94 (m, 2H);

¹³C NMR (125 MHz, DMSO-*d*₆) δ 168.73, 144.98, 128.66, 126.95, 126.75, 50.17, 44.66, 34.39, 33.51, 32.54, 26.56, 26.23, 26.09, 23.13.

HRMS (ESIpos) (*m/z*): calculated for C₁₆H₂₃NO 245.1774; found 245.1774.

N-(2-cyclopentyl-1-phenylethyl)acetamide (**6b**, unreported product)



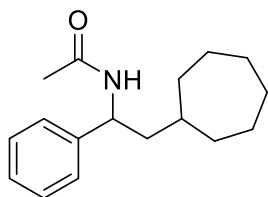
Following the **general procedure B**, white solid (97.0 mg, 84%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.29 (d, *J* = 8.6 Hz, 1H), 7.43–7.32 (m, 4H), 7.30–7.22 (m, 1H), 4.83 (td, *J* = 8.5, 6.0 Hz, 1H), 1.88 (s, 3H), 1.75 (ddq, *J* = 16.3, 11.4, 5.7 Hz, 4H), 1.62–1.57 (m, 3H), 1.55–1.43 (m, 2H), 1.24–1.09 (m, 2H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 168.71, 144.76, 128.65, 126.99, 126.84, 52.28, 43.39, 37.14, 32.62, 32.53, 25.19, 25.04, 23.16.

HRMS (ESIpos) (*m/z*): calculated for C₁₅H₂₁NO 231.1617; found 231.1617.

N-(2-cycloheptyl-1-phenylethyl)acetamide (**6c**, unreported product)



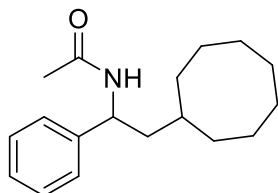
Following **the general procedure B**, white solid (111.6 mg, 86%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.28 (d, *J* = 8.6 Hz, 1H), 7.41–7.29 (m, 4H), 7.29–7.24 (m, 1H), 4.89 (td, *J* = 9.1, 5.6 Hz, 1H), 1.88 (s, 3H), 1.73–1.69 (m, 2H), 1.67–1.35 (m, 11H), 1.29–1.18 (m, 2H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 168.75, 144.94, 128.67, 126.95, 126.77, 50.75, 45.25, 35.73, 34.79, 33.67, 28.56, 28.53, 26.20, 26.08, 23.13.

HRMS (ESIpos) (*m/z*): calculated for C₁₇H₂₅NO 259.1930; found 259.1930.

N-(2-cyclooctyl-1-phenylethyl)acetamide (**6d**, unreported product)



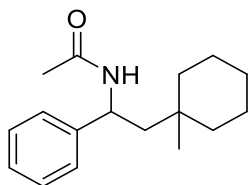
Following **the general procedure B**, white solid (107.5 mg, 80%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.24 (d, *J* = 8.6 Hz, 1H), 7.35–7.25 (m, 4H), 7.23–7.18 (m, 1H), 4.85 (td, *J* = 9.0, 4.9 Hz, 1H), 1.83 (s, 3H), 1.66–1.17 (m, 17H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 168.74, 144.94, 128.67, 126.96, 126.78, 50.69, 45.13, 33.61, 32.67, 31.03, 27.42, 27.19, 26.29, 25.31, 25.16, 23.12.

HRMS (ESIpos) (*m/z*): calculated for C₁₈H₂₇NO 273.2087; found 273.2087.

N-(2-(1-methylcyclohexyl)-1-phenylethyl)acetamide (**6e**, major product from the reaction with methylcyclohexane, unreported product)



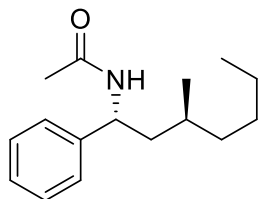
Following **the general procedure B**, white solid (44.0 mg, 34%).

$^1\text{H NMR}$ (600 MHz, $\text{DMSO-}d_6$) δ 8.25 (d, $J = 8.8$ Hz, 1H), 7.31–7.24 (m, 4H), 7.20–7.16 (m, 1H), 4.93 (td, $J = 9.0, 3.5$ Hz, 1H), 1.78 (s, 3H), 1.74 (dd, $J = 14.4, 9.1$ Hz, 1H), 1.48 (dd, $J = 14.4, 3.5$ Hz, 1H), 1.46–1.17 (m, 10H), 0.89 (s, 3H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 168.16, 146.39, 128.71, 126.80, 126.64, 49.17, 49.08, 38.11, 38.03, 33.32, 26.39, 25.56, 23.23, 21.99, 21.94.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{17}\text{H}_{25}\text{NO}$ 259.1930; found 259.1930.

N-((1*R*, 3*S*)-3-methyl-1-phenylheptyl)acetamide (**6f**, unreported product)



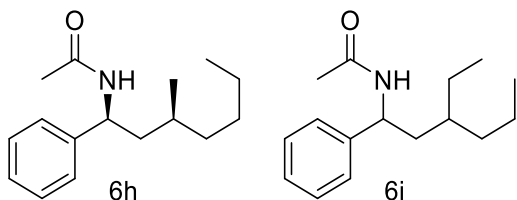
Following **the general procedure B**, colorless oil (5 mg, 4.1%).

$^1\text{H NMR}$ (600 MHz, $\text{DMSO-}d_6$) δ 8.23 (d, $J = 8.8$ Hz, 1H), 7.33–7.27 (m, 2H), 7.29–7.24 (m, 2H), 7.20 (tt, $J = 7.2, 1.5$ Hz, 1H), 4.83 (q, $J = 8.80, 8.5, 7.0$ Hz, 1H), 1.80 (s, 3H), 1.71–1.55 (m, 1H), 1.50–1.43 (m, 1H), 1.38–1.88 (m, 8H), 0.86–0.82 (m, 5H).

$^{13}\text{C NMR}$ (151 MHz, $\text{DMSO-}d_6$) δ 168.06, 144.05, 128.14, 126.50, 126.38, 50.33, 43.66, 35.20, 28.95, 28.18, 22.58, 22.21, 19.56, 13.87.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{16}\text{H}_{25}\text{NO}$ 247.1930; found 247.1931.

N-((1*S*,3*S*)-3-methyl-1-phenylheptyl)acetamide (**6g**, unreported product); *N*-(3-ethyl-1-phenylhexyl)acetamide (**6h**, unreported product)



6g and **6h** are a mixture (**6g**:**6h** = 2:2.3) as determined by $^1\text{H NMR}$.

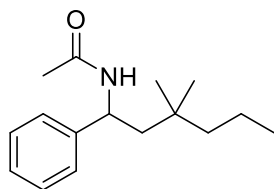
Following **the general procedure B**, colorless oil (94.0 mg, 76%).

¹H NMR (600 MHz, DMSO-*d*₆) δ 8.20 (dd, *J* = 8.7, 3.7 Hz, 1H), 7.32–7.23 (m, 4H), 7.19 (ddt, *J* = 8.7, 5.4, 1.6 Hz, 1H), 4.89–4.78 (m, 1H), 1.83–1.78 (m, 2H), 1.66 (ddd, *J* = 13.3, 10.5, 4.4 Hz, 0.37H), 1.56 (dp, *J* = 14.0, 4.3 Hz, 0.43H), 1.47 (dtd, *J* = 13.6, 6.3, 3.7 Hz, 0.38H), 1.41 (d, *J* = 5.5 Hz, 0.16H), 1.34–1.25 (m, 1H), 1.29–1.15 (m, 7H), 1.14–1.07 (m, 0.3H), 0.91–0.82 (m, 3H), 0.82–0.77 (m, 1H), 0.75 (t, *J* = 7.2 Hz, 1H).

¹³C NMR (151 MHz, DMSO-*d*₆) δ 168.83, 168.73, 168.69, 145.12, 144.86, 144.81, 128.66, 126.92, 126.74, 50.73, 50.67, 50.53, 44.54, 41.02, 40.99, 36.90, 35.37, 35.09, 35.02, 34.63, 29.68, 29.02, 25.88, 24.93, 23.10, 22.89, 19.51, 19.47, 19.21, 14.83, 14.72, 14.46, 10.87, 10.41.

HRMS (ESIpos) (*m/z*): calculated for C₁₆H₂₅NO 247.1930; found 247.1929 and 247.1930.

N-(3,3-dimethyl-1-phenylhexyl)acetamide (**6i**, Major product from the reaction with *iso*-hexane, unreported product)



Following the general procedure B, colorless oil (50.6 mg, 41%)

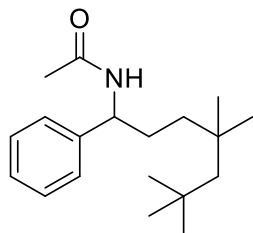
¹H NMR (600 MHz, DMSO-*d*₆) δ 8.23 (d, *J* = 8.6 Hz, 1H), 7.36–7.21 (m, 3H), 7.17 (ddt, *J* = 7.2, 5.3, 1.4 Hz, 1H), 4.89 (td, *J* = 9.1, 4.1 Hz, 1H), 1.78 (s, 3H), 1.72–1.65 (m, 1H), 1.45 (dd, *J* = 14.3, 3.6 Hz, 1H), 1.22–1.08 (m, 4H), 0.86–0.79 (m, 9H).

¹³C NMR (151 MHz, DMSO-*d*₆) δ 168.18, 146.24, 128.69, 126.81, 126.64, 49.60, 48.53, 44.83, 33.46, 27.91, 27.89, 23.21, 17.18, 15.33.

¹⁵N NMR (61 MHz, DMSO-*d*₆) δ -247.5.

HRMS (ESIpos) (*m/z*): calculated for C₁₆H₂₅NO 247.1930; found 247.1930.

N-(4,4,6,6-tetramethyl-1-phenylheptyl)acetamide (**6j**, unreported product)



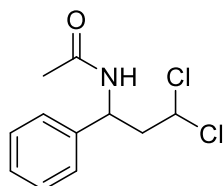
Following the general procedure B, colorless oil (40.4 mg, 28%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.28 (d, $J = 8.6$ Hz, 1H), 7.40–7.30 (m, 4H), 7.27 (t, $J = 7.0$ Hz, 1H), 4.72 (q, $J = 8.1$ Hz, 1H), 1.88 (s, 3H), 1.73–1.57 (m, 2H), 1.34 (ddd, $J = 13.4, 11.4, 5.5$ Hz, 1H), 1.26–1.10 (m, 3H), 1.02–0.84 (m, 15H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 186.76, 144.47, 128.62, 127.02, 126.93, 53.98, 53.75, 41.33, 34.65, 32.46, 32.28, 31.74, 29.42, 29.27, 23.14.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{19}\text{H}_{31}\text{NO}$ 289.2400; found 289.2401.

N-(3,3-dichloro-1-phenylpropyl)acetamide (**6k**, unreported product)



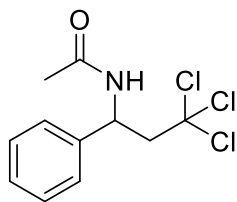
Following the general procedure B, white solid (53.9 mg, 45%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.42 (d, $J = 8.5$ Hz, 1H), 7.40–7.30 (m, 4H), 7.29–7.25 (m, 1H), 6.07 (dd, $J = 8.6, 4.5$ Hz, 1H), 5.02 (ddd, $J = 10.0, 8.4, 4.7$ Hz, 1H), 2.68–2.62 (m, 1H), 2.49–2.44 (m, 1H), 1.86 (s, 3H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 169.15, 142.44, 129.01, 127.71, 126.83, 72.30, 50.55, 49.79, 23.20.

HRMS (ESIpos) (m/z): $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{11}\text{H}_{13}\text{Cl}_2\text{NO}$ 246.0446; found 246.0447.

N-(3,3,3-trichloro-1-phenylpropyl)acetamide (**6l**, unreported product)



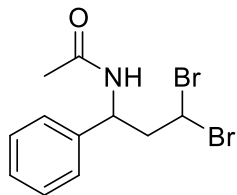
Following the general procedure B, white solid (102.8 mg, 74%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.60 (d, $J = 8.6$ Hz, 1H), 7.40–7.33 (m, 4H), 7.29–7.26 (m, 1H), 5.35 (td, $J = 8.4, 3.4$ Hz, 1H), 3.34 (dd, $J = 15.2, 3.4$ Hz, 1H), 3.09 (dd, $J = 15.2, 3.4$ Hz, 1H), 1.86 (s, 3H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 168.66, 142.75, 129.05, 127.76, 126.99, 98.40, 59.51, 51.02, 23.25.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{11}\text{H}_{12}\text{Cl}_3\text{NO}$ 278.9978; found 278.9977.

N-(3,3-dibromo-1-phenylpropyl)acetamide (**6m**, unreported product)



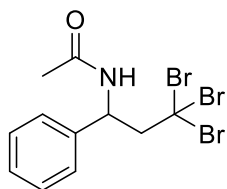
Following the general procedure B, colorless oil (54.7 mg, 33%).

$^1\text{H NMR}$ (600 MHz, $\text{DMSO-}d_6$) δ 8.36 (d, $J = 8.4$ Hz, 1H), 7.35–7.28 (m, 2H), 7.28–7.22 (m, 2H), 5.87 (dd, $J = 9.0, 4.7$ Hz, 1H), 4.94 (ddd, $J = 9.7, 8.3, 4.8$ Hz, 1H), 2.79 (ddd, $J = 14.6, 9.8, 4.7$ Hz, 1H), 2.63 (ddd, $J = 14.6, 9.0, 4.8$ Hz, 1H), 1.82 (s, 3H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 169.13, 142.31, 128.99, 127.67, 126.79, 52.09, 51.55, 44.73, 23.17.

HRMS (ESIpos) (m/z): $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{11}\text{H}_{13}\text{Br}_2\text{NONa}$ 355.9256; found 355.9253.

N-(3,3,3-tribromo-1-phenylpropyl)acetamide (**6n**, unreported product)



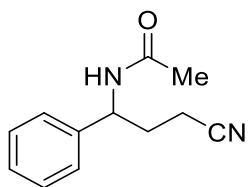
Following the general procedure B, colorless oil (49.2 mg, 24%) (Mixed with 21% of **6p**).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.68 (d, $J = 8.4$ Hz, 1H), 7.46–7.39 (m, 3H), 7.38–7.29 (m, 2H), 5.29 (ddd, $J = 9.7, 8.3, 4.8$ Hz, 1H), 3.71 (dd, $J = 15.4, 8.1$ Hz, 1H), 3.36 (d, $J = 2.8$ Hz, 1H), 1.92 (s, 3H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 186.61, 142.60, 128.52, 127.14, 126.34, 63.81, 53.44, 38.59, 23.37.

HRMS (ESIpos) (m/z): $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{11}\text{H}_{12}\text{Br}_3\text{NONa}$ 433.8361; found 433.8359.

N-(3-cyano-1-phenylpropyl)acetamide (**6o**, CAS: 2127514-83-2)



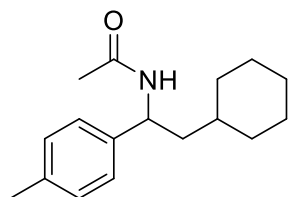
Following the general procedure B, white solid (86.8 mg, 86%).

$^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.38-7.35 (m, 2H), 7.32-7.30 (m, 1H), 7.27-7.26 (m, 2H), 5.90 (d, $J = 8.0$ Hz, 1H), 5.04 (dd, $J = 15.5$ Hz, 8.0 Hz, 1H), 2.37-2.31 (m, 2H), 2.27-2.22 (m, 1H), 2.15-2.10 (m, 1H), 1.99 (s, 3H).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 169.85, 139.83, 129.21, 128.34, 126.55, 119.26, 52.90, 31.66, 23.37, 14.54.

HRMS (ESIpos) (m/z): M^+ calculated for $\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}$ 202.1100; found 202.1098.

N-(2-cyclohexyl-1-(*p*-tolyl)ethyl)acetamide (**7a**, unreported product)



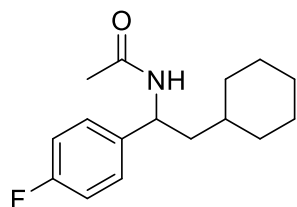
Following **the general procedure B**, colorless oil (103.6 mg, 80%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.16 (d, $J = 8.6$ Hz, 1H), 7.20-7.07 (m, 4H), 4.82 (td, $J = 9.0, 6.1$ Hz, 1H), 2.27 (s, 3H), 1.81 (s, 3H), 1.77-1.49 (m, 6H), 1.43 (ddd, $J = 13.8, 7.9, 6.1$ Hz, 1H), 1.26-1.06 (m, 4H), 0.97-0.81 (m, 2H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 168.61, 141.89, 135.93, 129.20, 126.70, 49.85, 44.62, 34.37, 33.44, 32.63, 26.56, 26.22, 26.10, 23.14, 21.09.

HRMS (ESIpos) (m/z): $[M+\text{Na}]^+$ calculated for $\text{C}_{11}\text{H}_{13}\text{Br}_2\text{NONa}$ 333.9436; found 333.9434.

N-(2-cyclohexyl-1-(4-fluorophenyl)ethyl)acetamide (**7c**, unreported product)



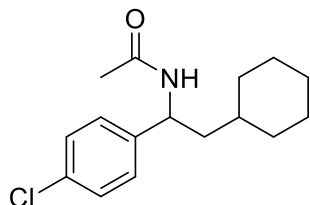
Following **the general procedure B**, colorless oil (65.7 mg, 50%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.28 (d, $J = 8.5$ Hz, 1H), 7.49-7.30 (m, 2H), 7.18 (t, $J = 8.9$ Hz, 2H), 4.91 (td, $J = 9.1, 5.9$ Hz, 1H), 1.88 (s, 3H), 1.84-1.55 (m, 6H), 1.49 (ddd, $J = 13.8, 8.1, 5.9$ Hz, 1H), 1.3-1.11 (m, 4H), 1.02-0.88 (m, 2H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 168.76, 141.14 (d, $J = 3.75$ Hz, 1C), 128.65 (d, $J = 6.25$ Hz, 1C), 115.44, 115.27, 49.55, 44.53, 34.37, 33.44, 32.52, 26.55, 26.22, 26.07, 23.12.

HRMS (ESIpos) (m/z): calculated for C₁₂H₂₂FNO 263.1679; found 263.1681.

N-(1-(4-chlorophenyl)-2-cyclohexylethyl)acetamide (**7d**, unreported product)



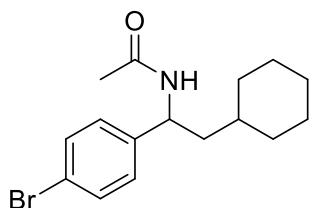
Following **the general procedure B**, colorless oil (113.0 mg, 81%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.31 (d, *J* = 8.4 Hz, 1H), 7.56–7.11 (m, 4H), 4.90 (td, *J* = 9.1, 5.9 Hz, 1H), 1.88 (s, 3H), 1.84–1.55 (m, 6H), 1.48 (ddd, *J* = 13.8, 8.1, 5.9 Hz, 1H), 1.32–1.12 (m, 4H), 1.04–0.85 (m, 2H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 168.85, 144.01, 131.46, 128.67, 128.63, 49.68, 44.33, 34.35, 33.44, 32.49, 26.54, 26.21, 26.06, 23.10.

HRMS (ESIpos) (m/z): [M+Na]⁺ calculated for C₁₆H₂₂ClNONa 302.1282; found 302.1278.

N-(1-(4-bromophenyl)-2-cyclohexylethyl)acetamide (**7e**, unreported product)



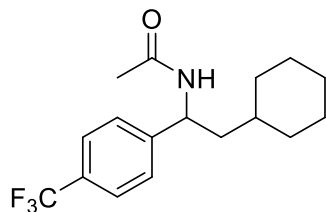
Following **the general procedure B**, colorless oil (127.6 mg, 79%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.31 (d, *J* = 8.4 Hz, 1H), 7.55 (d, *J* = 8.5 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 4.88 (td, *J* = 9.1, 5.9 Hz, 1H), 1.88 (s, 3H), 1.82–1.57 (m, 6H), 1.48 (ddd, *J* = 13.8, 8.1, 5.9 Hz, 1H), 1.36–1.06 (m, 4H), 1.03–0.81 (m, 2H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 168.86, 144.45, 131.55, 129.07, 119.93, 49.75, 44.28, 34.34, 33.44, 32.49, 26.53, 26.20, 26.06, 23.09.

HRMS (ESIpos) (m/z): calculated for C₁₆H₂₂BrNO 323.0879; found 323.0883.

N-(2-cyclohexyl-1-(4-(trifluoromethyl)phenyl)ethyl)acetamide (**7f**, unreported product)



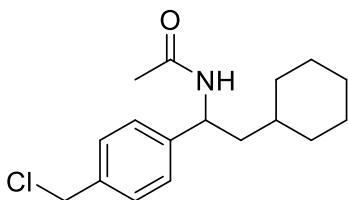
Following the general procedure B, colorless oil (95.5 mg, 61%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.35 (d, *J* = 8.2 Hz, 1H), 7.68 (d, *J* = 8.1 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 2H), 4.92 (td, *J* = 9.2, 5.6 Hz, 1H), 1.84 (s, 3H), 1.78–1.54 (m, 6H), 1.45 (ddd, *J* = 13.8, 8.3, 5.6 Hz, 1H), 1.31–1.08 (m, 4H), 0.99–0.78 (m, 2H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 169.05, 127.57, 127.54, 125.63 (q, *J* = 30, 15 Hz, 1C), 50.11, 44.22, 34.33, 33.50, 32.28, 26.53, 26.20, 26.03, 23.06.

HRMS (ESIpos) (*m/z*): calculated for C₁₇H₂₂F₃NO 312.1580; found 312.1584.

N-(1-(4-(chloromethyl)phenyl)-2-cyclohexylethyl)acetamide (**7g**, unreported product)



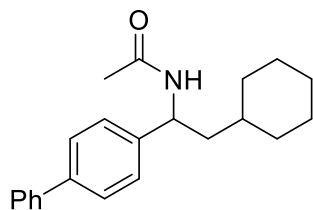
Following the general procedure B, colorless oil (102.5 mg, 70%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.29 (d, *J* = 8.5 Hz, 1H), 7.42 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 8.2 Hz, 2H), 4.91 (td, *J* = 9.1, 6.0 Hz, 1H), 4.79 (s, 2H), 1.88 (s, 3H), 1.82–1.59 (m, 6H), 1.50 (ddd, *J* = 13.8, 8.1, 5.9 Hz, 1H), 1.35–1.14 (m, 4H), 1.04–0.89 (m, 2H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 168.82, 145.25, 136.30, 129.29, 127.06, 49.97, 46.53, 44.45, 34.36, 33.47, 32.52, 26.55, 26.21, 26.07, 23.11.

HRMS (ESIpos) (*m/z*): calculated for C₁₇H₂₄ClNO 293.1540; found 293.1542.

N-(1-([1,1'-biphenyl]-4-yl)-2-cyclohexylethyl)acetamide (**7h**, unreported product)



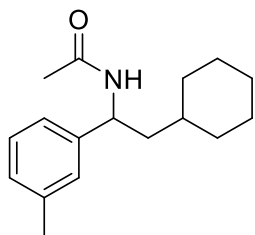
Following **the general procedure B**, white solid (101.1 mg, 63%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.32 (d, $J = 8.5$ Hz, 1H), 7.78–7.61 (m, 3H), 7.52 (t, $J = 7.7$ Hz, 2H), 7.42 (dd, $J = 7.9, 2.7$ Hz, 3H), 4.96 (td, $J = 9.1, 5.9$ Hz, 1H), 1.90 (s, 3H), 1.84–1.62 (m, 6H), 1.55 (ddd, $J = 13.8, 8.1, 5.9$ Hz, 1H), 1.38–1.12 (m, 4H), 1.06–0.91 (m, 2H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 168.80, 144.24, 140.50, 138.95, 129.37, 127.73, 127.39, 127.06, 49.92, 44.51, 34.40, 33.51, 32.56, 26.57, 26.23, 26.09, 23.16.

HRMS (ESIpos) (m/z): $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{22}\text{H}_{27}\text{NONa}$ 344.1984; found 344.1987.

N-(2-cyclohexyl-1-(*m*-tolyl)ethyl)acetamide (**7i**, unreported product)



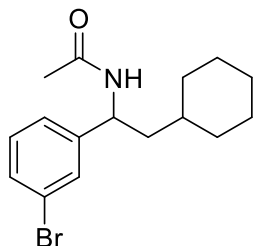
Following **the general procedure B**, colorless oil (97.1 mg, 75%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.23 (d, $J = 8.6$ Hz, 1H), 7.24 (d, $J = 7.6$ Hz, 1H), 7.18–6.77 (m, 3H), 4.88 (td, $J = 9.2, 5.7$ Hz, 1H), 2.34 (s, 3H), 1.87 (s, 3H), 1.82–1.57 (m, 6H), 1.48 (ddd, $J = 13.8, 8.2, 5.7$ Hz, 1H), 1.32–1.14 (m, 4H), 1.04–0.86 (m, 2H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 168.69, 144.95, 137.66, 128.57, 127.59, 127.38, 123.84, 50.09, 44.69, 34.39, 33.54, 32.51, 26.57, 26.23, 26.09, 23.15, 21.57.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{17}\text{H}_{25}\text{NO}$ 259.1930; found 259.1930.

N-(1-(3-bromophenyl)-2-cyclohexylethyl)acetamide (**7j**, unreported product)



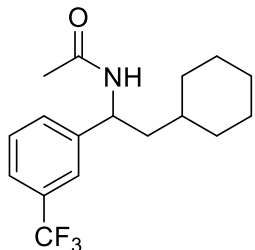
Following the general procedure B, colorless oil (133.8 mg, 83%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.33 (d, $J = 8.5$ Hz, 1H), 7.52 (d, $J = 1.3$ Hz, 1H), 7.46 (ddd, $J = 5.7, 3.6, 2.0$ Hz, 1H), 7.36–7.30 (m, 2H), 5.15–4.60 (m, 1H), 1.89 (s, 3H), 1.82–1.59 (m, 6H), 1.48 (ddd, $J = 13.7, 8.3, 5.5$ Hz, 1H), 1.33–1.13 (m, 4H), 1.06–0.88 (m, 2H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 168.96, 147.97, 130.94, 129.90, 129.41, 126.00, 122.11, 49.89, 44.40, 34.38, 33.51, 32.37, 26.54, 26.22, 26.05, 23.10.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{16}\text{H}_{22}\text{BrNO}$ 323.0879; found 323.0881.

N-(2-cyclohexyl-1-(3-(trifluoromethyl)phenyl)ethyl)acetamide (**7k**, unreported product)



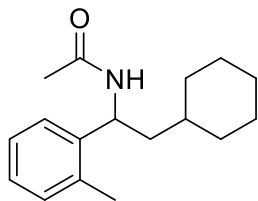
Following the general procedure B, colorless oil (70.4 mg, 45%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.41 (d, $J = 8.4$ Hz, 1H), 7.72–7.57 (m, 4H), 5.00 (ddd, $J = 10.1, 8.3, 5.4$ Hz, 1H), 1.90 (s, 3H), 1.82–1.62 (m, 6H), 1.50 (ddd, $J = 13.8, 8.5, 5.4$ Hz, 1H), 1.37–1.13 (m, 4H), 1.05–0.89 (m, 2H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 169.07, 146.63, 131.05, 129.82, 123.82 (d, $J = 4.25$ Hz, 1C), 123.09 (q, $J = 364, 17$ Hz, 1C), 50.02, 44.40, 34.41, 33.54, 32.31, 26.53, 26.22, 26.05, 23.08.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{17}\text{H}_{22}\text{F}_3\text{NO}$ 313.1647; found 313.1649.

N-(2-cyclohexyl-1-(*o*-tolyl)ethyl)acetamide (**7l**, unreported product)



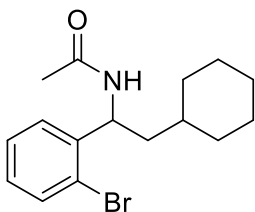
Following the general procedure B, colorless oil (117.8 mg, 91%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.30 (d, *J* = 8.4 Hz, 1H), 7.40–7.31 (m, 1H), 7.21 (td, *J* = 8.0, 3.0 Hz, 1H), 7.19–7.11 (m, 2H), 5.15 (ddd, *J* = 10.4, 8.2, 3.7 Hz, 1H), 2.36 (s, 3H), 1.87 (s, 3H), 1.75–1.49 (m, 5H), 1.46–1.34 (m, 2H), 1.26–1.16 (m, 3H), 1.03–0.96 (m, 2H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 168.73, 143.57, 134.54, 130.38, 126.67, 126.50, 125.75, 46.29, 44.07, 34.56, 33.92, 32.25, 26.56, 26.27, 26.08, 23.08, 19.18.

HRMS (ESIpos) (*m/z*): calculated for C₁₇H₂₅NO 259.1930; found 259.1931.

N-(1-(2-bromophenyl)ethyl)cyclohexylacetamide (**7m**, unreported product)



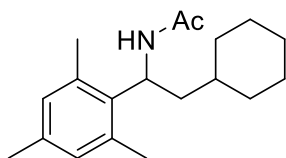
Following the general procedure B, colorless oil (129.2 mg, 80%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.45 (d, *J* = 8.3 Hz, 1H), 7.60 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.49–7.38 (m, 2H), 7.21 (ddd, *J* = 8.0, 7.0, 2.0 Hz, 1H), 5.66–5.04 (m, 1H), 1.92–1.90 (m, 4H), 1.78–1.61 (m, 4H), 1.53–1.41 (m, 3H), 1.31–1.16 (m, 3H), 1.00 (t, *J* = 6.9 Hz, 2H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 169.06, 144.45, 132.90, 128.93, 128.44, 127.71, 122.38, 49.87, 43.68, 34.58, 34.01, 31.90, 26.54, 26.25, 26.02, 23.06.

HRMS (ESIpos) (*m/z*): [M+Na]⁺ calculated for C₁₆H₂₂BrNONa 346.0777; found 346.0776.

N-(2-cyclohexyl-1-mesitylethyl)acetamide (**7n**, unreported product)



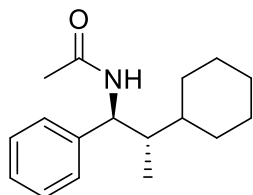
Following **the general procedure B**, white solid (88.9 mg, 62%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.11 (d, *J* = 7.1 Hz, 1H), 6.77 (s, 2H), 5.21 (ddd, *J* = 11.2, 7.1, 4.4 Hz, 1H), 2.38 (s, 6H), 2.21 (s, 3H), 1.95–1.88 (m, 1H), 1.86 (s, 3H), 1.83–1.62 (m, 6H), 1.46–1.12 (m, 5H), 1.08–0.84 (m, 2H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 168.88, 137.59, 135.63, 135.09, 47.06, 40.91, 34.52, 34.06, 32.13, 26.58, 26.30, 26.10, 22.80, 21.00, 20.73.

HRMS (ESIpos) (*m/z*): calculated for C₁₉H₂₉NO 287.2243; found 287.2242.

N-((1*S*, 2*S*)-1,2-diphenylpropyl)acetamide (1*S*, 2*S*-**7o**, unreported product)



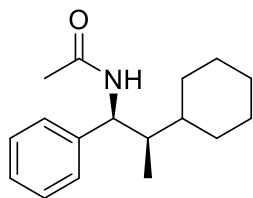
Following **the general procedure B**, colorless oil (33.0 mg, 25.5 %).

¹H NMR (600 MHz, DMSO-*d*₆) δ 8.26 (d, *J* = 9.2 Hz, 1H), 7.31–7.27 (m, 2H), 7.26–7.23 (m, 2H), 7.22–7.18 (m, 1H), 4.59 (dd, *J* = 10.6, 9.2 Hz, 1H), 1.79 (s, 3H), 1.77–1.54 (m, 5H), 1.47 (d, *J* = 12.4 Hz, 1H), 1.33–1.06 (m, 5H), 0.91 (td, *J* = 12.4, 8.9 Hz, 1H), 0.45 (d, *J* = 7.0 Hz, 3H).

¹³C NMR (151 MHz, DMSO-*d*₆) δ 168.41, 144.04, 128.52, 127.85, 127.01, 55.58, 42.81, 38.38, 32.10, 27.15, 26.80, 26.78, 26.11, 23.11, 12.53.

HRMS (ESIpos) (*m/z*): calculated for C₁₇H₂₅NO 259.1930; found 259.1931.

N-((1*S*, 2*R*)-2-cyclohexyl-1-phenylpropyl)acetamide (1*S*, 2*R*-**7o**, unreported product)



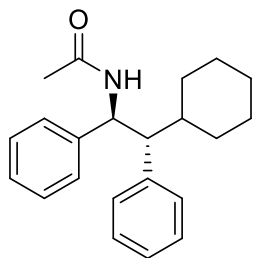
Following **the general procedure B**, colorless oil (33.0 mg, 25.5 %)

¹H NMR (600 MHz, DMSO-*d*₆) δ 8.07 (d, *J* = 9.2 Hz, 1H), 7.33–7.28 (m, 2H), 7.25–7.16 (m, 3H), 4.88 (dd, *J* = 9.3, 7.6 Hz, 1H), 1.85 (s, 3H), 1.73–1.59 (m, 4H), 1.57–1.49 (m, 2H), 1.15–0.86 (m, 5H), 0.77 (d, *J* = 6.9 Hz, 3H).

¹³C NMR (151 MHz, DMSO-*d*₆) δ 168.96, 143.90, 128.56, 127.21, 126.86, 54.87, 43.59, 39.13, 31.61, 27.99, 26.73, 26.59, 26.50, 23.11, 11.77.

HRMS (ESIpos) (m/z): calculated for C₁₇H₂₅NO 259.1930; found 259.1931.

N-((1*S*, 2*R*)-2-cyclohexyl-1,2-diphenylethyl)acetamide (1*S*, 2*R*-**7p**, unreported product, minor component)



Following the general procedure B, colorless oil (21.3 mg, 13.3%).

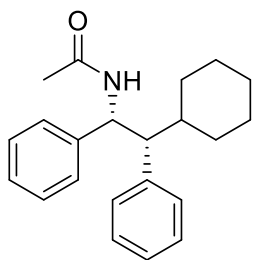
¹H NMR (600 MHz, DMSO-*d*₆) δ 8.35 (d, *J* = 9.3 Hz, 1H), 7.36–6.96 (m, 10H), 5.38 (dd, *J* = 9.3 Hz, 11.7 Hz, 1H), 3.02 (dd, *J* = 3.9 Hz, 11.7 Hz, 1H), 1.91–1.81 (m, 5H), 1.67–1.32 (m, 2H), 1.58–1.50 (m, 2H), 1.24–1.22 (m, 1H), 1.11–1.08 (m, 1H), 0.95–0.58 (m, 3H).

¹³C NMR (151 MHz, DMSO-*d*₆) δ 168.47, 143.51, 139.61, 130.28, 128.16, 128.10, 127.75, 126.62, 126.30, 55.85, 53.16, 39.00, 32.60, 27.35, 26.91, 26.75, 26.59, 23.18.

HRMS (ESIpos) (m/z): [M+H]⁺ calculated for C₂₂H₂₇NO 322.2165; found 322.2165.

N-((1*S*, 2*S*)-2-cyclohexyl-1,2-diphenylethyl)acetamide (1*S*, 2*S*-**7p**, unreported product, major component)

Following the general procedure B, colorless oil (42.6 mg, 26.7%).

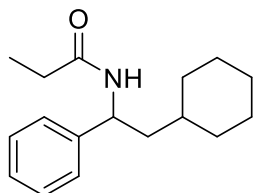


¹H NMR (600 MHz, DMSO-*d*₆) δ 7.94 (d, *J* = 9.3 Hz, 1H), 7.36–6.96 (m, 10H), 5.37 (dd, *J* = 9.3 Hz, 10.9 Hz, 1H), 2.91 (dd, *J* = 4.3 Hz, 10.9 Hz, 1H), 1.75–1.71 (m, 1H), 1.58–1.47 (m, 6H), 1.43–1.42 (m, 1H), 1.14–1.11 (m, 1H), 0.95–0.58 (m, 5H).

¹³C NMR (151 MHz, DMSO-*d*₆) δ 168.21, 143.61, 139.73, 129.92, 128.67, 127.75, 127.68, 127.27, 126.62, 126.54, 56.73, 53.23, 38.61, 32.67, 27.64, 26.59, 26.38, 26.37, 22.96.

HRMS (ESIpos) (m/z): [M+H]⁺ calculated for C₂₂H₂₇NO 322.2165; found 322.2165.

N-(2-cyclohexyl-1-phenylethyl)propionamide (**8a**, unreported product)



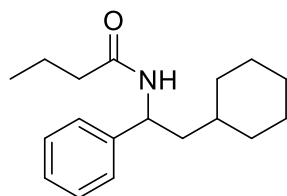
Following the general procedure C, white solid (90.6 mg, 70%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.13 (d, *J* = 8.6 Hz, 1H), 7.51–7.24 (m, 4H), 7.23–7.17 (m, 1H), 4.88 (td, *J* = 9.2, 5.8 Hz, 1H), 2.10 (m, 2H), 1.77 – 1.54 (m, 6H), 1.45 (m, 1H), 1.29–1.07 (m, 4H), 0.98 (t, *J* = 7.6 Hz, 3H), 0.94–0.80 (m, 1H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 172.53, 145.09, 128.66, 126.91, 126.71, 50.00, 44.66, 34.47, 33.53, 32.47, 29.04, 26.56, 26.26, 26.13, 10.57.

HRMS (ESIpos) (m/z): calculated for C₁₇H₂₅NO 259.1930; found 259.1933.

N-(2-cyclohexyl-1-phenylethyl)butyramide (**8b**, unreported product)



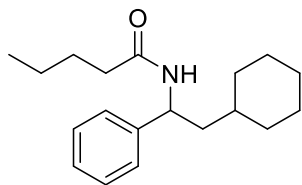
Following the general procedure C, white solid (79.1 mg, 58%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.16 (d, *J* = 8.7 Hz, 1H), 7.35–7.24 (m, 4H), 7.23–7.17 (m, 1H), 4.89 (td, *J* = 9.5, 5.4 Hz, 1H), 2.09–2.05 (m, 2H), 1.66–1.43 (m, 9H), 1.24–1.11 (m, 4H), 0.94–0.81 (m, 3H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 171.64, 145.16, 128.64, 126.90, 126.69, 49.91, 44.61, 37.85, 34.49, 33.63, 32.31, 26.55, 26.32, 26.13, 19.29, 13.95.

HRMS (ESIpos) (m/z): calculated for C₁₈H₂₇NO 273.2087; found 273.2088.

N-(2-cyclohexyl-1-phenylethyl)pentanamide (**8c**, unreported product)



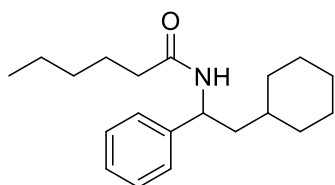
Following the general procedure C, white solid (86.1 mg, 60%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.16 (d, $J = 8.6$ Hz, 1H), 7.34–7.24 (m, 4H), 7.23–7.18 (m, 1H), 4.89 (td, $J = 9.5, 5.4$ Hz, 1H), 2.19–2.02 (m, 2H), 1.74 (d, $J = 12.8$ Hz, 1H), 1.69–1.37 (m, 8H), 1.28–1.22 (m, 3H), 1.15–1.09 (m, 3H), 1.04–0.86 (m, 2H), 0.85 (t, $J = 7.4$ Hz, 3H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 171.77, 145.16, 128.64, 126.89, 126.67, 49.90, 44.63, 35.60, 34.49, 33.65, 32.31, 28.01, 26.56, 26.13, 22.11, 14.16.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{19}\text{H}_{29}\text{NO}$ 287.2243; found 287.2242.

N-(2-cyclohexyl-1-phenylethyl)hexanamide (**8d**, unreported product)



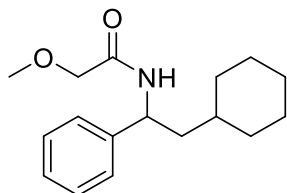
Following the general procedure C, white solid (114.4 mg, 76%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.16 (d, $J = 8.6$ Hz, 1H), 7.32–7.25 (m, 4H), 7.23–7.17 (m, 1H), 4.89 (td, $J = 9.6, 5.3$ Hz, 1H), 2.15–2.03 (m, 2H), 1.75 (d, $J = 13.0$ Hz, 1H), 1.71–1.35 (m, 8H), 1.37–1.05 (m, 8H), 1.00–0.88 (m, 1H), 0.85 (t, $J = 7.2$ Hz, 4H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 171.78, 145.18, 128.62, 126.89, 126.67, 49.87, 44.64, 35.83, 34.49, 33.68, 32.28, 31.21, 26.56, 26.35, 26.14, 25.56, 22.32, 14.37.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{20}\text{H}_{31}\text{NO}$ 301.2400; found 301.2400.

N-(2-cyclohexyl-1-phenylethyl)-2-methoxyacetamide (**8f**, unreported product)



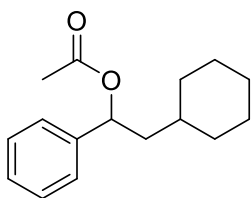
Following the general procedure C, white solid (61.7 mg, 48%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.14 (d, $J = 8.8$ Hz, 1H), 7.31 (d, $J = 6.7$ Hz, 4H), 7.25–7.18 (m, 1H), 4.95 (td, $J = 9.4, 5.7$ Hz, 1H), 3.81 (d, $J = 3.8$ Hz, 2H), 3.30 (s, 3H), 1.78–1.37 (m, 7H), 1.29–1.06 (m, 4H), 0.98–0.83 (m, 2H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 168.67, 144.63, 128.67, 127.07, 126.93, 71.99, 58.95, 49.85, 43.92, 34.44, 33.50, 32.40, 26.56, 26.25, 26.10.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{17}\text{H}_{25}\text{NO}_2$ 275.1879; found 275.1879.

2-cyclohexyl-1-phenylethyl acetate (**8g**, CAS: 2366999-88-2)



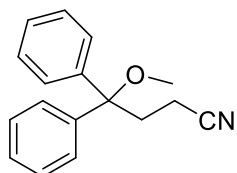
Following the general procedure C, colorless oil (77.5 mg, 63%).

$^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 7.46–7.30 (m, 5H), 5.80 (dd, $J = 8.8, 5.6$ Hz, 1H), 2.09 (s, 3H), 1.83 (ddd, $J = 14.4, 8.8, 6.1$ Hz, 1H), 1.78–1.57 (m, 6H), 1.31–1.14 (m, 4H), 1.03–0.96 (m, 2H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ 170.23, 141.58, 128.88, 128.16, 126.67, 73.61, 43.94, 34.08, 33.33, 32.75, 26.45, 26.11, 26.01, 21.41.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{16}\text{H}_{22}\text{O}_2$ 246.1614; found 246.1616.

4-methoxy-4,4-diphenylbutanenitrile (**8h**, CAS: 1653998-30-1)



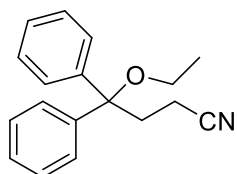
Following the procedure C, pale yellow solid (64.3 mg, 51%).

¹H NMR (500 MHz, CDCl₃) δ 7.33–7.29 (m, 8H), 7.25–7.21 (m, 2H), 3.05 (s, 3H), 2.79–2.62 (m, 2H), 2.25–2.03 (m, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 143.40, 128.32, 127.38, 126.76, 120.10, 81.46, 50.37, 31.34, 11.52.

HRMS (ESIpos) (m/z): calculated for C₁₇H₁₇NO 252.1382; found 252.1381.

4-ethoxy-4,4-diphenylbutanenitrile (**8i**, CAS: 1808942-62-2)



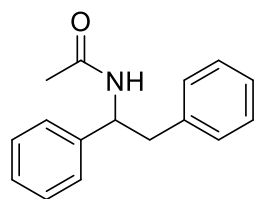
Following the procedure C, yellow oil (54.5 mg, 41%) .

¹H NMR (500 MHz, CDCl₃) δ 7.33–7.28 (m, 8H), 7.25–7.21 (m, 2H), 3.15 (q, *J* = 6.9 Hz, 2H), 2.73–2.66 (m, 2H), 2.17–2.10 (m, 2H), 1.21 (t, *J* = 6.9 Hz, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 143.89, 128.27, 127.27, 126.64, 120.18, 81.01, 57.84, 32.02, 15.36, 11.59.

HRMS (ESIpos) (m/z): calculated for C₁₈H₂₀NO 266.1539; found 266.1544.

N-(1,2-diphenylethyl)acetamide (**10a**, CAS: 21511-90-0)



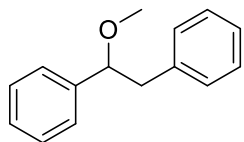
Following the general procedure D, white solid (118.3 mg, 99%).

¹H NMR (500 MHz, DMSO-*d*₆) δ 8.42 (d, *J* = 8.7 Hz, 1H), 7.41–7.34 (m, 4H), 7.33–7.20 (m, 6H), 5.08 (td, *J* = 8.6, 6.6 Hz, 1H), 3.07–2.87 (m, 2H), 1.82 (s, 3H).

¹³C NMR (125 MHz, DMSO-*d*₆) δ 168.73, 143.89, 139.19, 129.52, 128.61, 128.46, 127.17, 127.04, 126.53, 54.50, 42.75, 23.08.

HRMS (ESIpos) (m/z): calculated for C₁₆H₁₇NO 240.1382; found 240.1386.

(1-methoxyethane-1,2-diyl)dibenzene (**10b**, CAS: 27820-29-7)



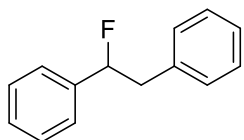
Following the general procedure D, colorless oil (33 mg, 31%) .

$^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.34 (ddd, $J = 8.8, 6.4, 0.9$ Hz, 2H), 7.29–7.25 (m, 3H), 7.23 (dd, $J = 7.9, 6.5$ Hz, 2H), 7.20–7.10 (m, 3H), 4.40 (dd, $J = 7.9, 5.7$ Hz, 1H), 3.07 (s, 3H), 3.02 (dd, $J = 13.8, 7.9$ Hz, 1H), 2.85 (dd, $J = 13.8, 5.6$ Hz, 1H).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 142.01, 138.90, 129.78, 128.68, 128.39, 127.96, 127.19, 126.43, 84.32, 5650. 44.17.

HRMS (ESIpos) (m/z): $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{15}\text{H}_{16}\text{ONa}$ 235.1093; found 235.1094.

(1-fluoroethane-1,2-diyl)dibenzene (**10c**, CAS: 74185-77-6)



Colorless solid (26.0 mg, 25%)

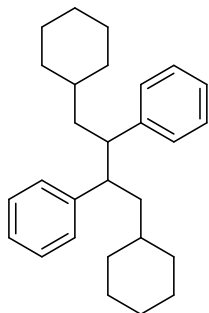
$^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.42–7.21 (m, 8H), 7.20–7.15 (m, 2H), 5.61 (ddd, $J = 47.4, 8.1, 4.8$ Hz, 1H), 3.27 (ddd, $J = 17.5, 14.3, 8.1$ Hz, 1H), 3.11 (ddd, $J = 28.6, 14.3, 4.8$ Hz, 1H).

$^{19}\text{F NMR}$ (471 MHz, CDCl_3) δ -153.64 – -193.14 (m).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 129.53, 128.40, 128.38, 128.36, 126.70, 125.70, 125.65, 94.90 (d, $J = 172.5$ Hz, 1C), 43.96 (d, $J = 23.8$ Hz, 1C).

HRMS (ESIpos) (m/z): calculated for $\text{C}_{14}\text{H}_{13}\text{F}$ 200.0995; found 200.0995.

(1,4-dicyclohexylbutane-2,3-diyl)dibenzene (**9**, CAS: 644985-99-9)



Colorless solid (48.6 mg, 31%)

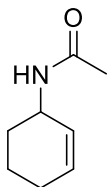
$^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.29 (t, $J = 7.5$ Hz, 4H), 7.23–7.17 (m, 2H), 7.16–7.10 (m, 4H), 2.79–2.72 (m, 2H), 1.70 (d, $J = 12.4$ Hz, 2H), 1.52–1.39 (m, 7H), 1.31 (dtd, $J = 12.9, 10.7, 10.1, 3.1$ Hz, 4H), 1.08–1.03 (m, 2H), 0.98–0.93 (m, 5H), 0.76–0.66 (m, 4H), 0.51 (qd, $J = 11.8, 3.7$ Hz, 2H).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 145.02, 128.36, 128.12, 125.82, 49.37, 42.33, 34.58, 34.49, 31.69, 26.57, 26.16, 25.94.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{28}\text{H}_{38}$ 374.2968; found 374.2964.

N-(cyclohex-2-en-1-yl)acetamide (**11a**, CAS: 39819-72-2)

Following the general procedure E, colorless solid (49.6 mg, 70%)

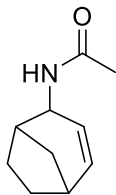


$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 5.81–5.85 (m, 1H), 5.53–5.48(m, 1H), 4.45–4.39 (m, 1H), 1.96–1.90 (m, 2H), 1.90 (s, 3H), 1.89–1.79 (m, 1H), 1.61–1.53 (m, 2H), 1.49–1.40 (m, 1H).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 169.16, 130.94, 127.64, 44.68, 29.43, 24.79, 23.51, 19.69.

HRMS (ESIpos) (m/z): calculated for $\text{C}_8\text{H}_{13}\text{NO}$ 140.1069; found 140.1068.

N-(bicyclo[3.2.1]oct-3-en-2-yl)acetamide (**11b**, CAS: 1823085-93-3)



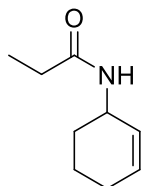
Following **the general procedure E**, colorless solid (59.4 mg, 72%)

$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 6.05–5.99 (m, 1H), 5.47 (s, 1H), 5.27–5.22 (m, 1H), 4.06–4.02 (m, 1H), 2.42–2.33 (m, 2H), 1.90 (s, 3H), 1.83–1.81 (m, 1H), 1.60–1.53 (m, 2H), 1.44–1.22 (m, 3H).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 168.64, 138.76, 123.26, 52.31, 38.50, 35.41, 32.19, 31.52, 26.63, 23.52.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{10}\text{H}_{15}\text{NO}$ 165.1148; found 165.1149.

N-(cyclohex-2-en-1-yl)propionamide (**11c**, CAS: 95973-99-2)



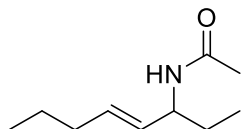
Following **the general procedure E**, colorless solid (25.2 mg, 33%)

$^1\text{H NMR}$ (300 MHz, $\text{DMSO}-d_6$) δ 7.81 (d, $J = 8.0$ Hz, 1H), 5.83–5.81 (m, 1H), 5.57–5.49 (m, 1H), 4.30–4.27 (m, 1H), 2.11 (q, $J = 7.5, 3.5$ Hz, 2H), 2.02–1.99 (m, 2H), 1.80–1.1.76 (m, 2H), 1.60–1.56 (m, 1H), 1.48–1.44 (m, 1H), 1.03 (t, $J = 7.5$ Hz, 1H).

$^{13}\text{C NMR}$ (125 MHz, $\text{DMSO}-d_6$) δ 172.57, 129.56, 129.35, 44.26, 29.48, 28.87, 24.86, 20.18, 10.45.

HRMS (ESIpos) (m/z): calculated for $\text{C}_9\text{H}_{15}\text{NO}$ 153.1148; found 153.1149.

(*E*)-*N*-(oct-4-en-3-yl)acetamide (**11d**, CAS: 2308508-60-1)



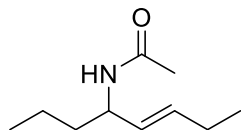
Following **the general procedure E**, colorless solid (41.4 mg, 49%)

$^1\text{H NMR}$ (600 MHz, $\text{DMSO}-d_6$) δ 7.78 (d, $J = 10.8$ Hz, 1H), 5.55–5.49 (m, 1H), 5.39–5.35 (m, 1H), 4.17–4.11 (m, 1H), 2.03–1.99 (m, 2H), 1.86 (s, 3H), 1.46–1.37 (m, 4H), 0.91 (t, $J = 9.0$ Hz, 3H), 0.86 (t, $J = 9.0$ Hz, 3H).

^{13}C NMR (125 MHz, DMSO- d_6) δ 168.61, 131.58, 130.09, 51.99, 34.19, 28.21, 23.17, 22.38, 13.94, 10.86.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{10}\text{H}_{19}\text{NO}$ 169.1461; found 169.1461.

(*E*)-*N*-(oct-5-en-4-yl)acetamide (**11e**, CAS: 2308508-61-2)



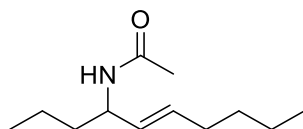
Following the general procedure E, colorless solid (33.8 mg, 40%)

^1H NMR (600 MHz, DMSO- d_6) δ 7.77 (d, J = 10.2 Hz, 1H), 5.59–5.54 (m, 1H), 5.39–5.34 (m, 1H), 4.26–4.21 (m, 1H), 2.07–2.01 (m, 2H), 1.85 (s, 3H), 1.44–1.40 (m, 2H), 1.32–1.27 (m, 2H), 0.99 (t, J = 9.0 Hz, 3H), 0.90 (t, J = 8.4 Hz, 3H).

^{13}C NMR (125 MHz, DMSO- d_6) δ 168.52, 131.54, 130.70, 50.04, 37.48, 25.11, 23.17, 19.18, 14.19, 13.99.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{10}\text{H}_{19}\text{NO}$ 169.1461; found 169.1461.

(*E*)-*N*-(dec-5-en-4-yl)acetamide (**11f**, unreported product)



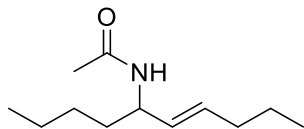
Following the general procedure E, colorless solid (36.4 mg, 37%)

^1H NMR (600 MHz, DMSO- d_6) δ 7.71 (d, J = 6.0 Hz, 1H), 5.46–5.41 (m, 1H), 5.32–5.27 (m, 1H), 4.18–4.11 (m, 1H), 1.95–1.91 (m, 2H), 1.78 (s, 3H), 1.38–1.30 (m, 2H), 1.28–1.18 (m, 5H), 0.86–0.82 (m, 7H).

^{13}C NMR (125 MHz, DMSO- d_6) δ 167.98, 131.17, 129.46, 49.56, 36.92, 31.18, 30.85, 22.62, 21.84, 18.61, 13.70, 13.64.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{12}\text{H}_{23}\text{NO}$ 197.3172; found 197.3171.

(*E*)-*N*-(dec-6-en-5-yl)acetamide (**11g**, CAS: 131317-75-4)



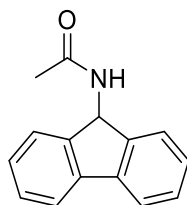
Following the general procedure E, colorless solid (36.4 mg, 37%)

$^1\text{H NMR}$ (600 MHz, DMSO- d_6) δ 7.71 (d, J = 6.0 Hz, 1H), 5.46–5.41 (m, 1H), 5.32–5.27 (m, 1H), 4.18–4.11 (m, 1H), 1.95–1.91 (m, 2H), 1.78 (s, 3H), 1.38–1.30 (m, 2H), 1.28–1.18 (m, 5H), 0.86–0.82 (m, 7H).

$^{13}\text{C NMR}$ (125 MHz, DMSO- d_6) δ 167.98, 131.40, 129.30, 49.87, 34.45, 33.62, 27.65, 22.62, 21.82, 21.48, 13.86, 13.38.

HRMS (ESIpos) (m/z): calculated for $\text{C}_{12}\text{H}_{23}\text{NO}$ 197.3172; found 197.3171.

N-(9H-fluoren-9-yl)acetamide (**13a**, CAS: 5424-77-1)



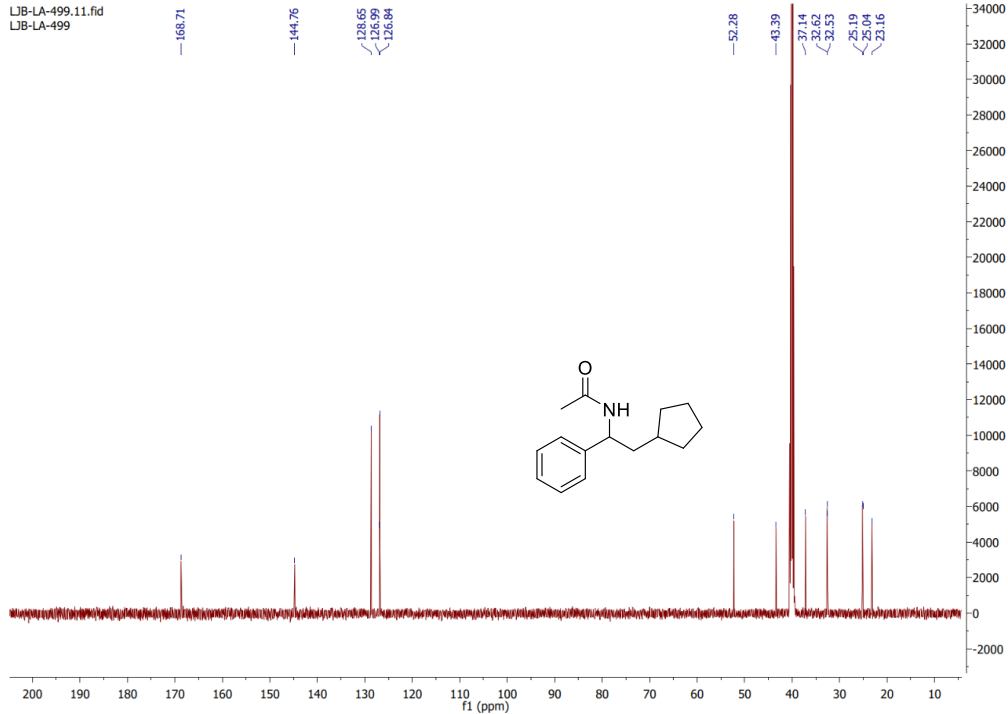
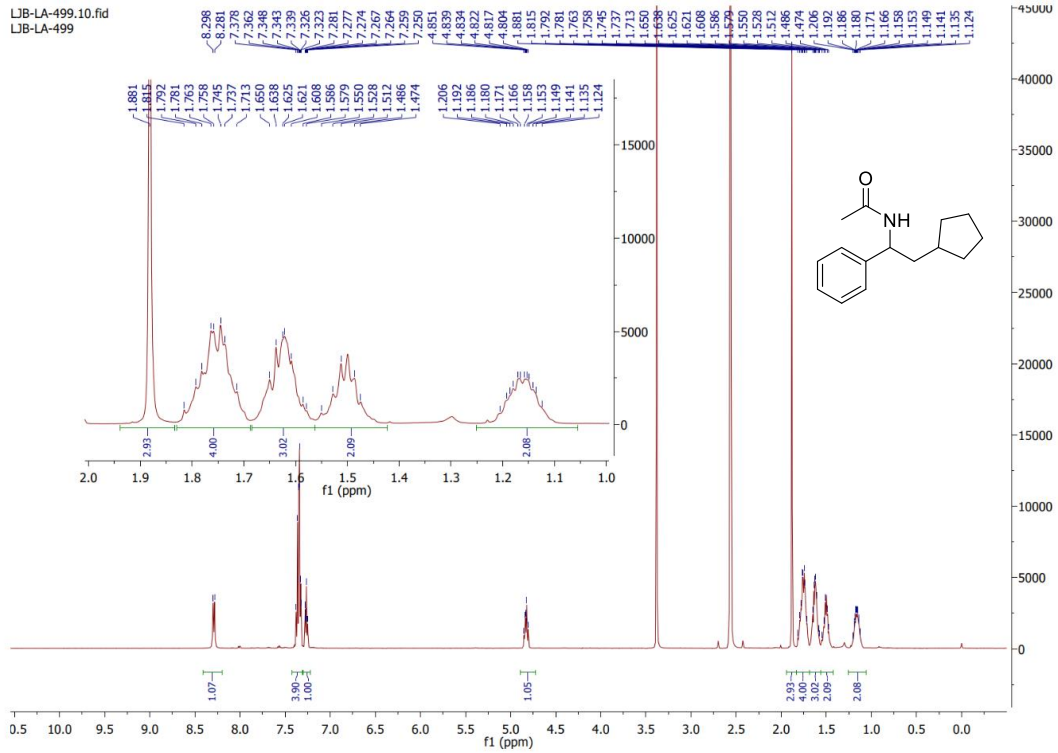
Colorless solid (78.1 mg, 70%)

$^1\text{H NMR}$ (300 MHz, DMSO- d_6) δ 8.50 (d, J = 8.4 Hz, 1H), 7.92–7.89 (m, 2 H), 7.57–7.47 (m, 2H), 7.46–7.45 (m, 2H), 7.41–7.36 (m, 2H), 6.07 (d, J = 8.4 Hz, 1H), 2.0 (s, 3H).

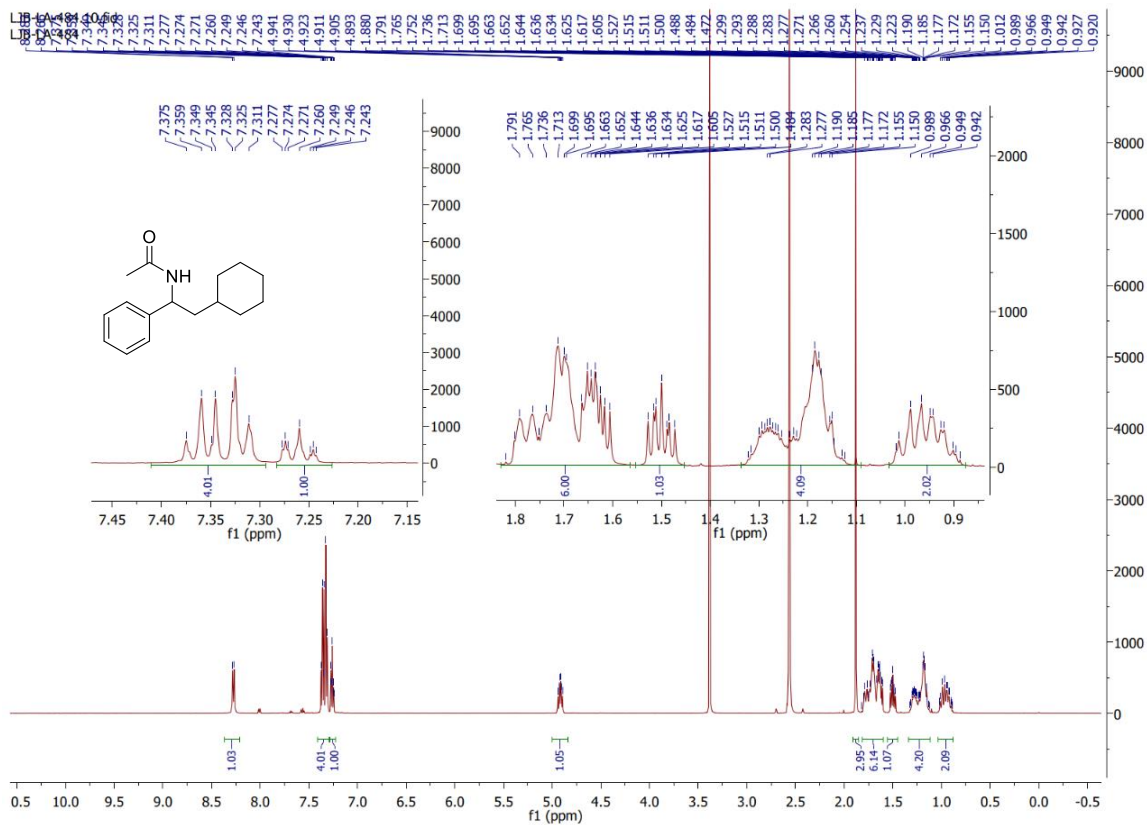
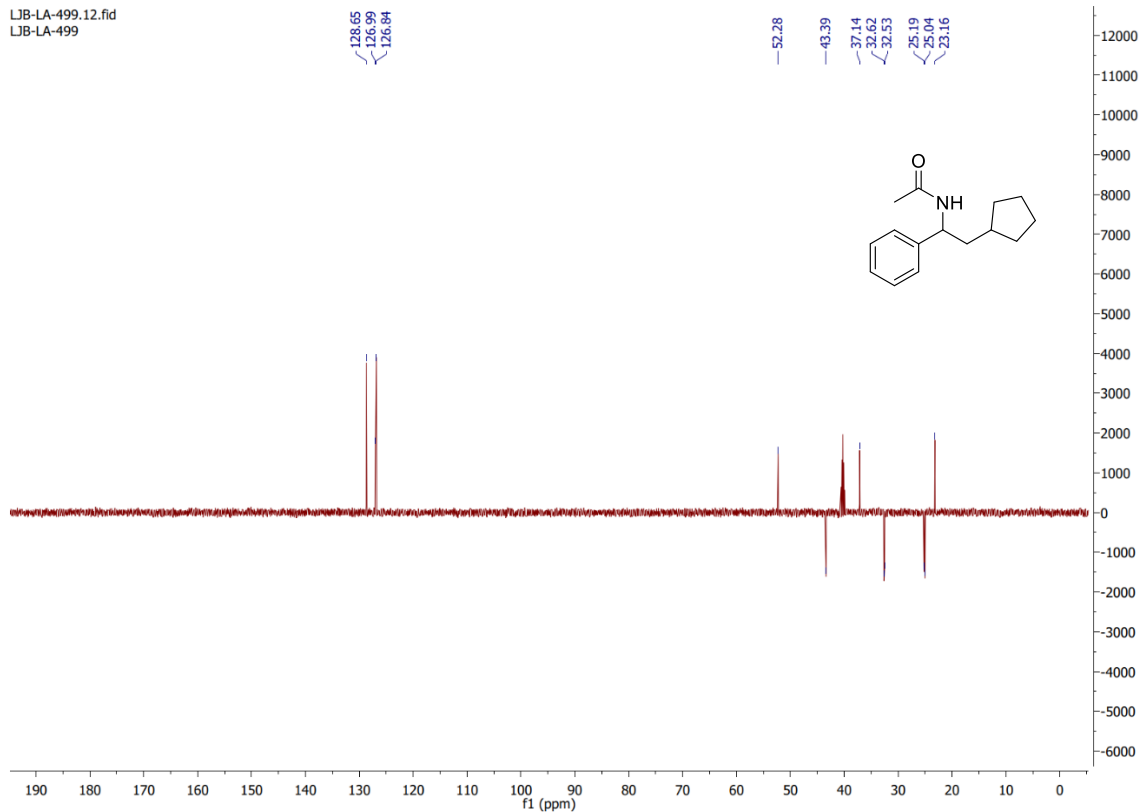
$^{13}\text{C NMR}$ (125 MHz, DMSO- d_6) δ 170.07, 144.86, 139.95, 128.28, 127.54, 124.82, 120.05, 54.02, 22.53.

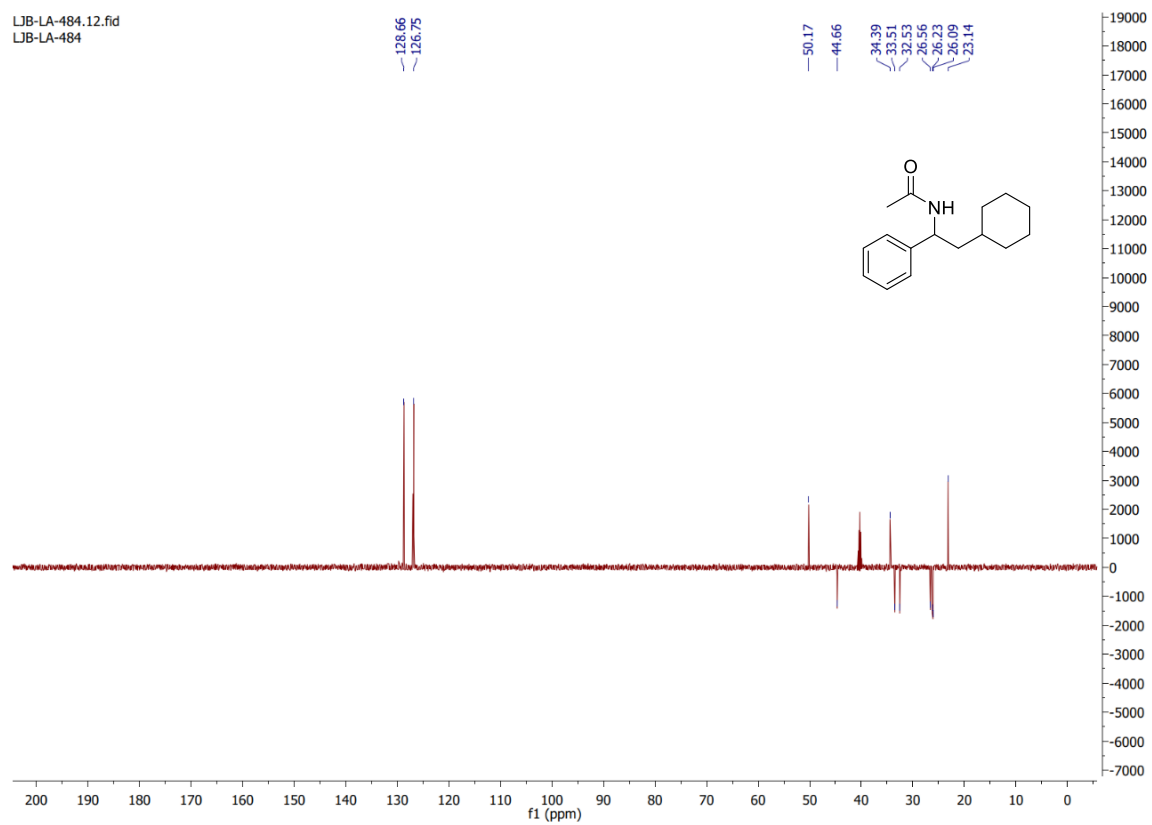
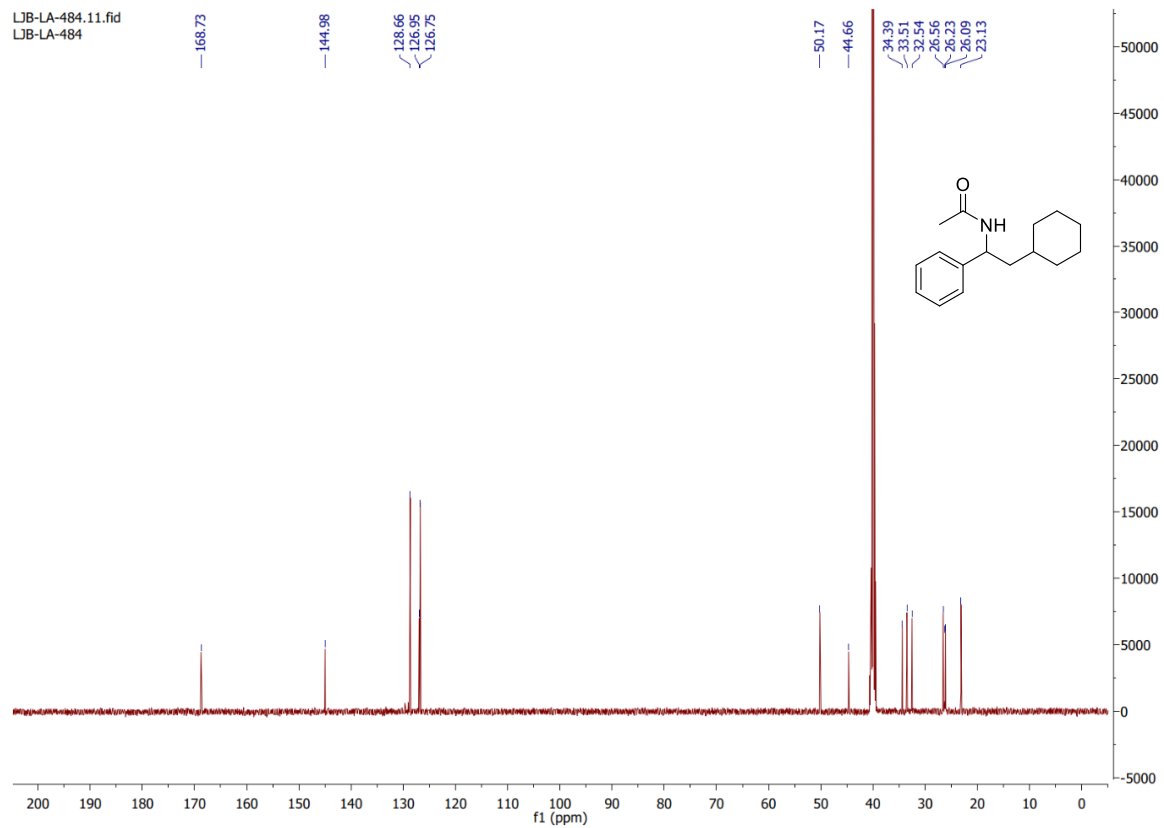
HRMS (ESIpos) (m/z): calculated for $\text{C}_{15}\text{H}_{13}\text{NO}$ 223.0989; found 223.0997.

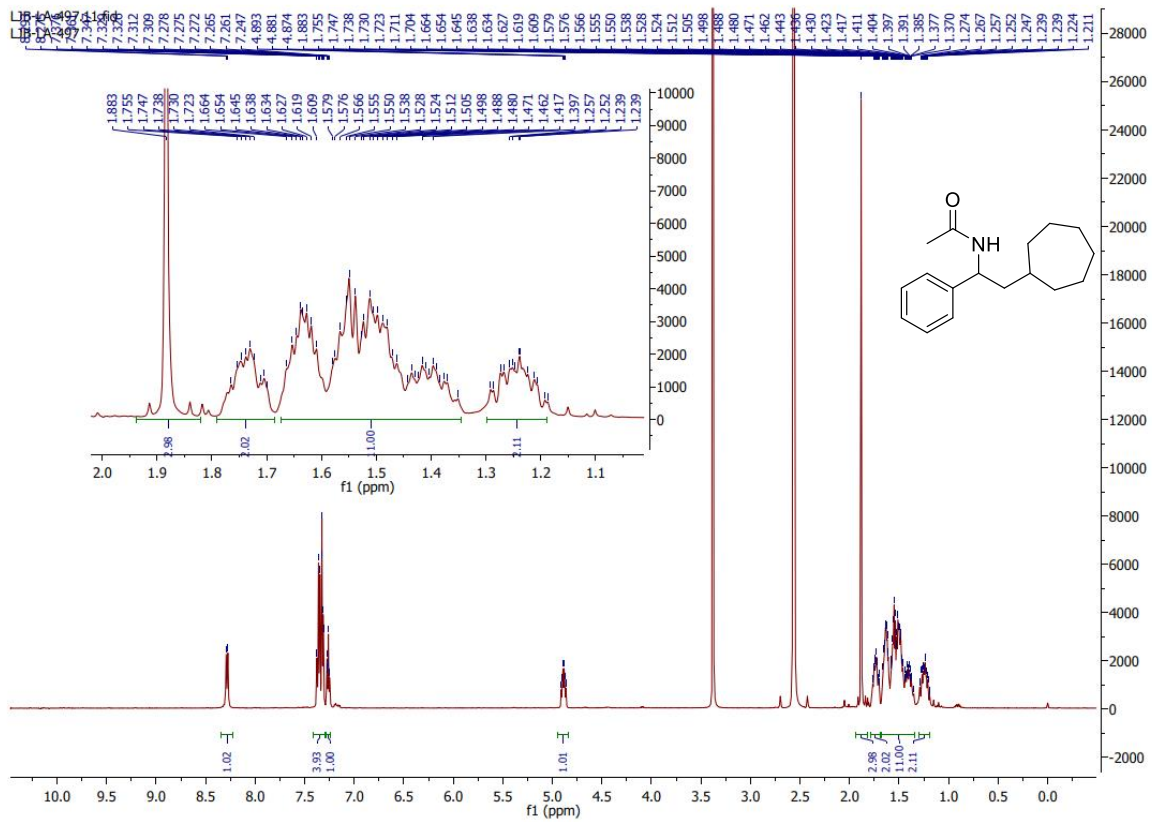
NMR and some HRMS Spectra of the Products

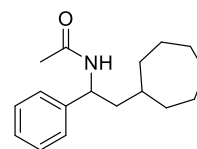
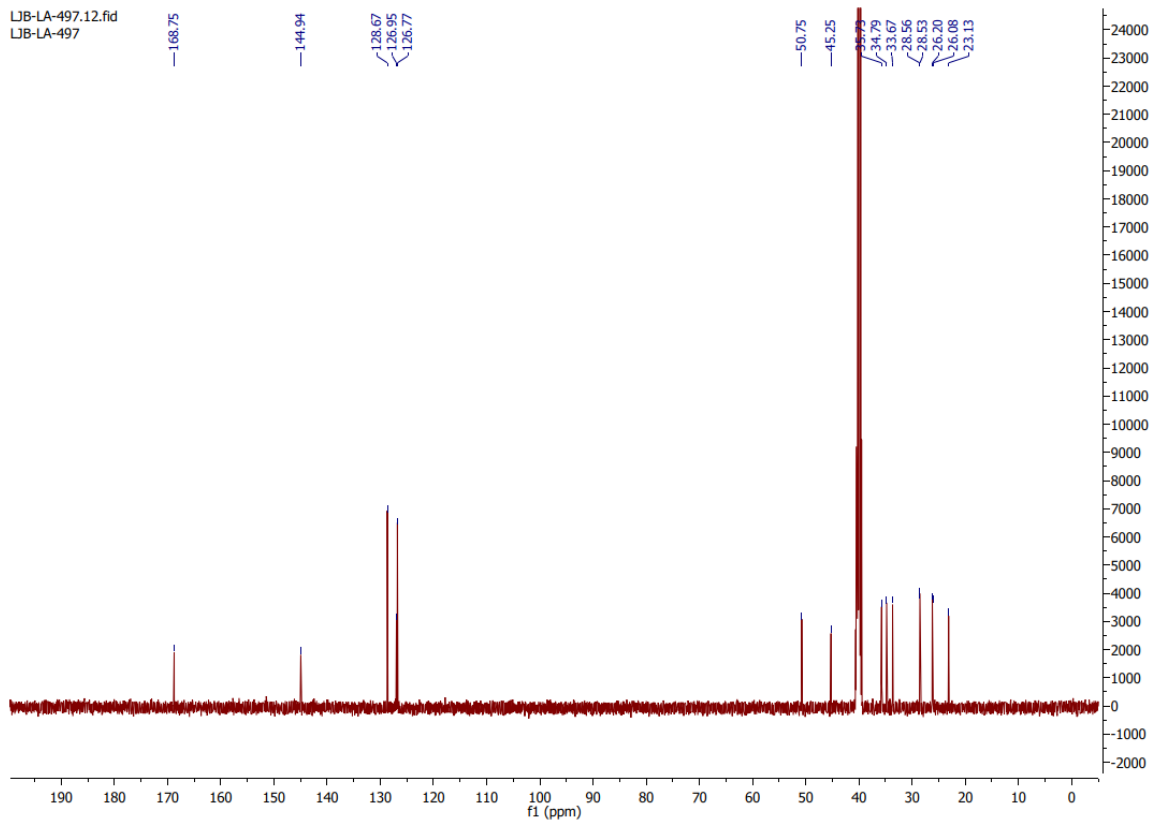


LJB-LA-499.12.fid
LJB-LA-499

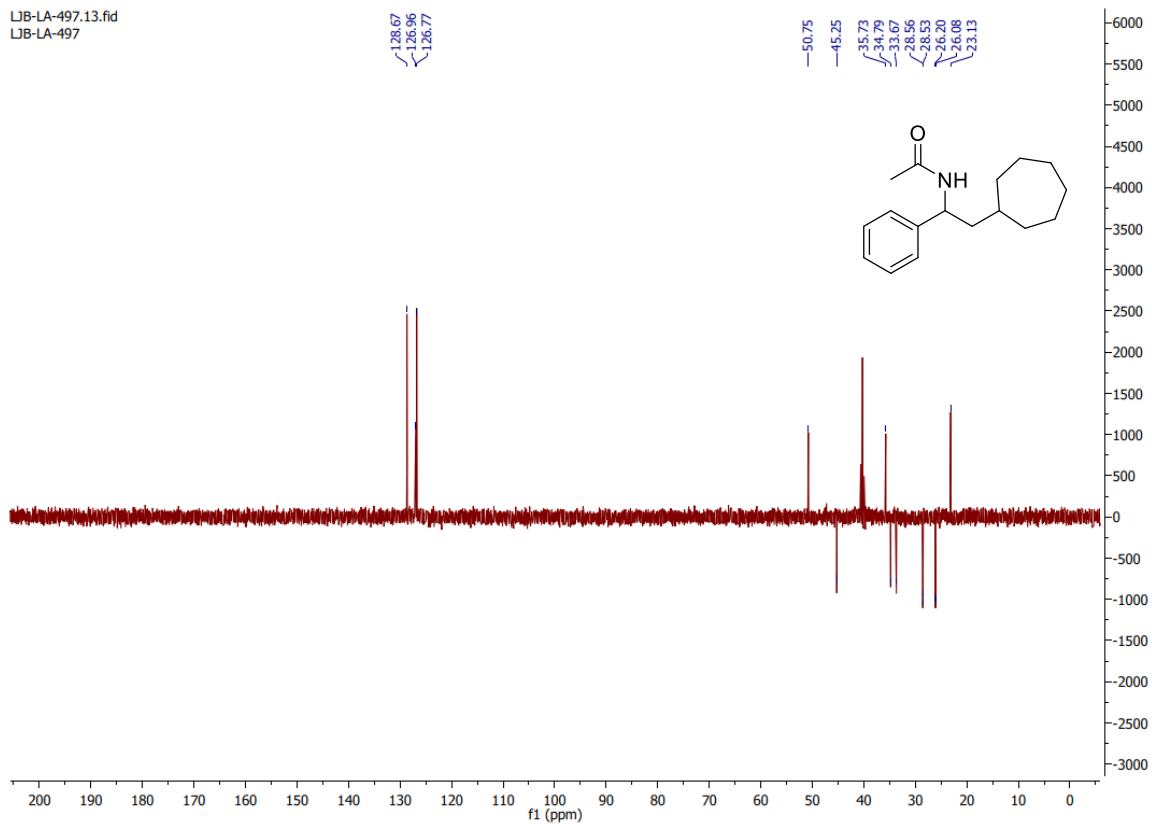




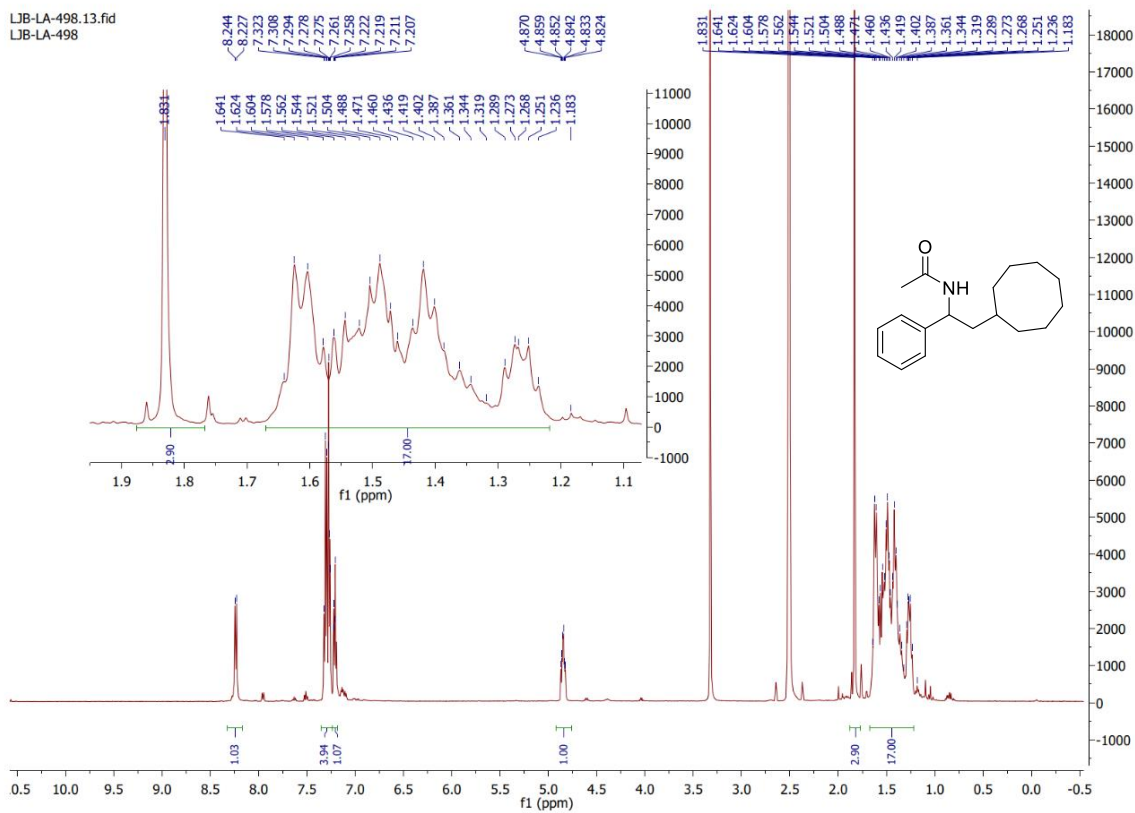


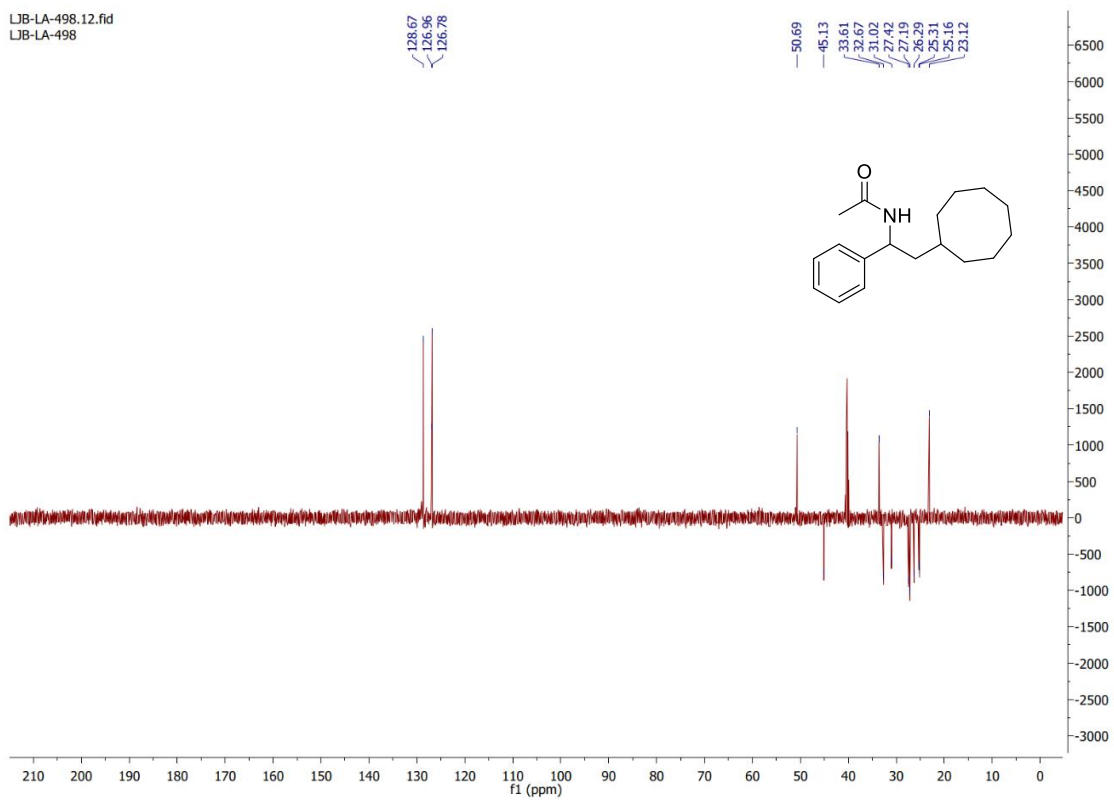
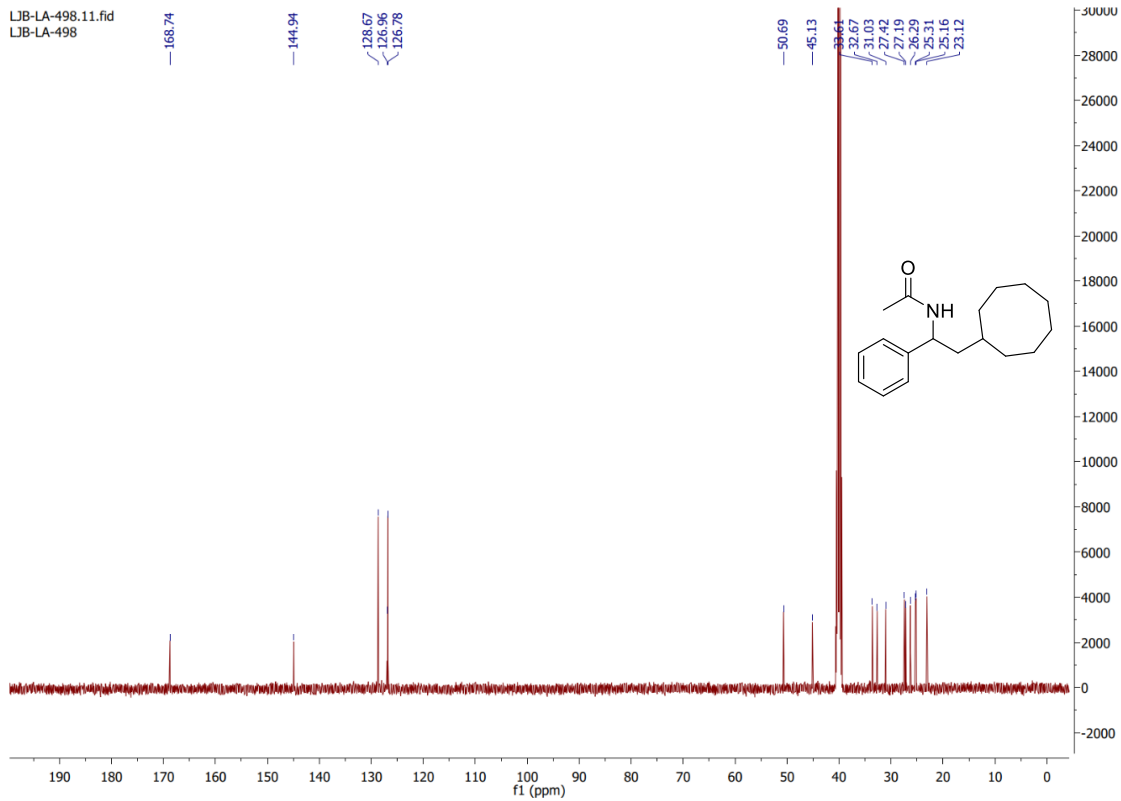


LJB-LA-497.13.fid
LJB-LA-497

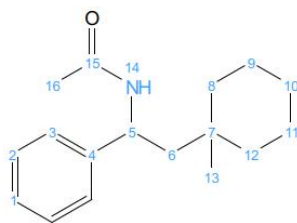


LJB-LA-498.13.fid
LJB-LA-498





LJB-LA-511
CDCl₃; 298 K; AV600a



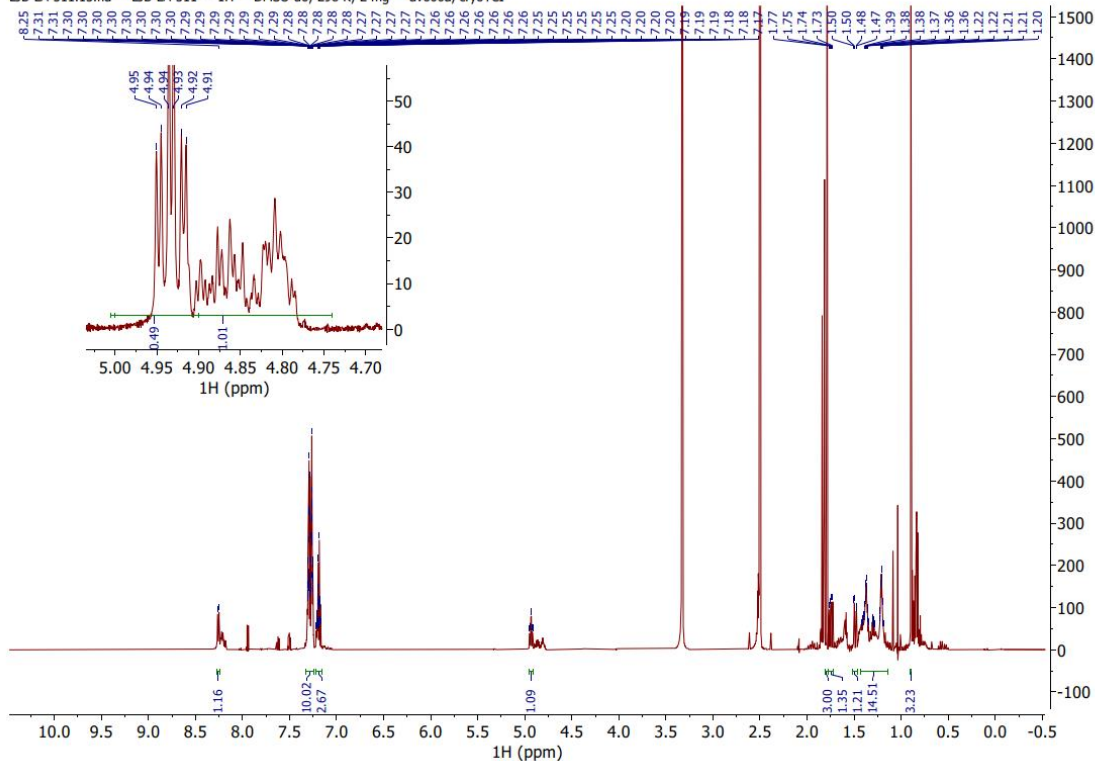
Major compound

C9, 10 and 11 are possibly to be interchanged. H8-H12 are overlapped at 1.46-1.17 ppm.

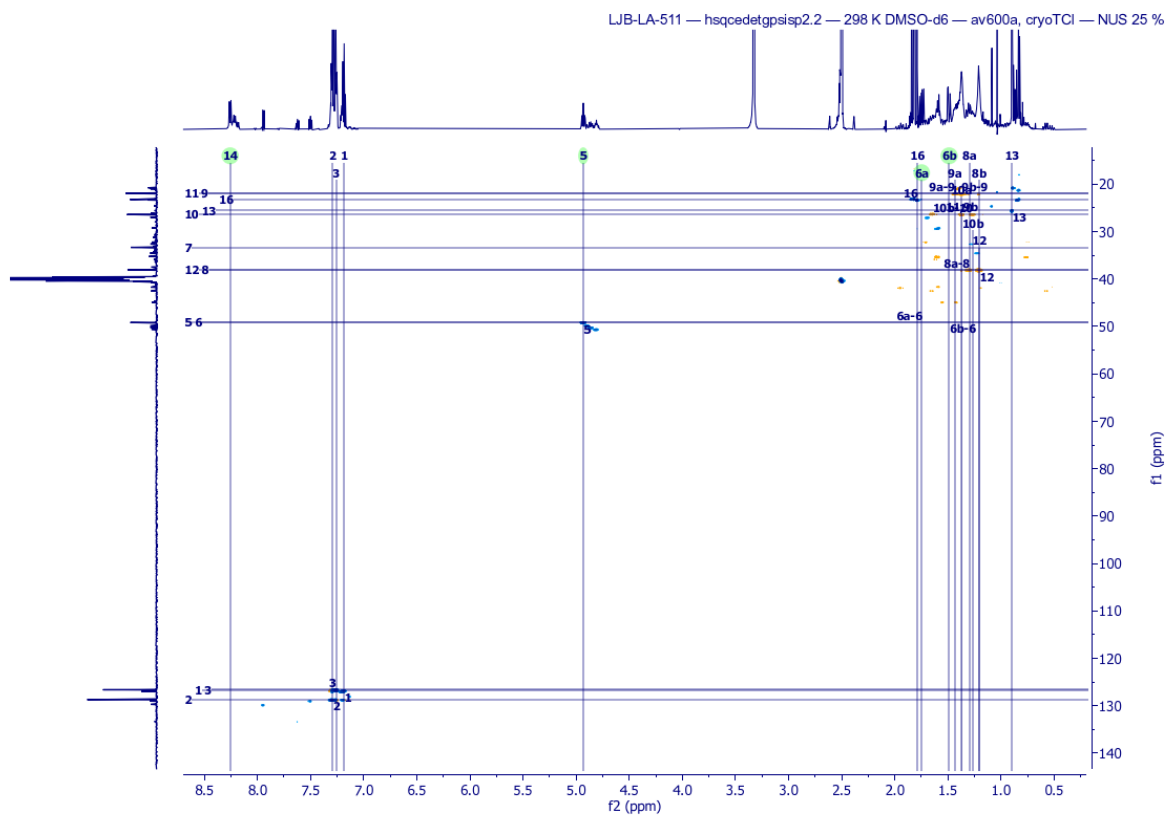
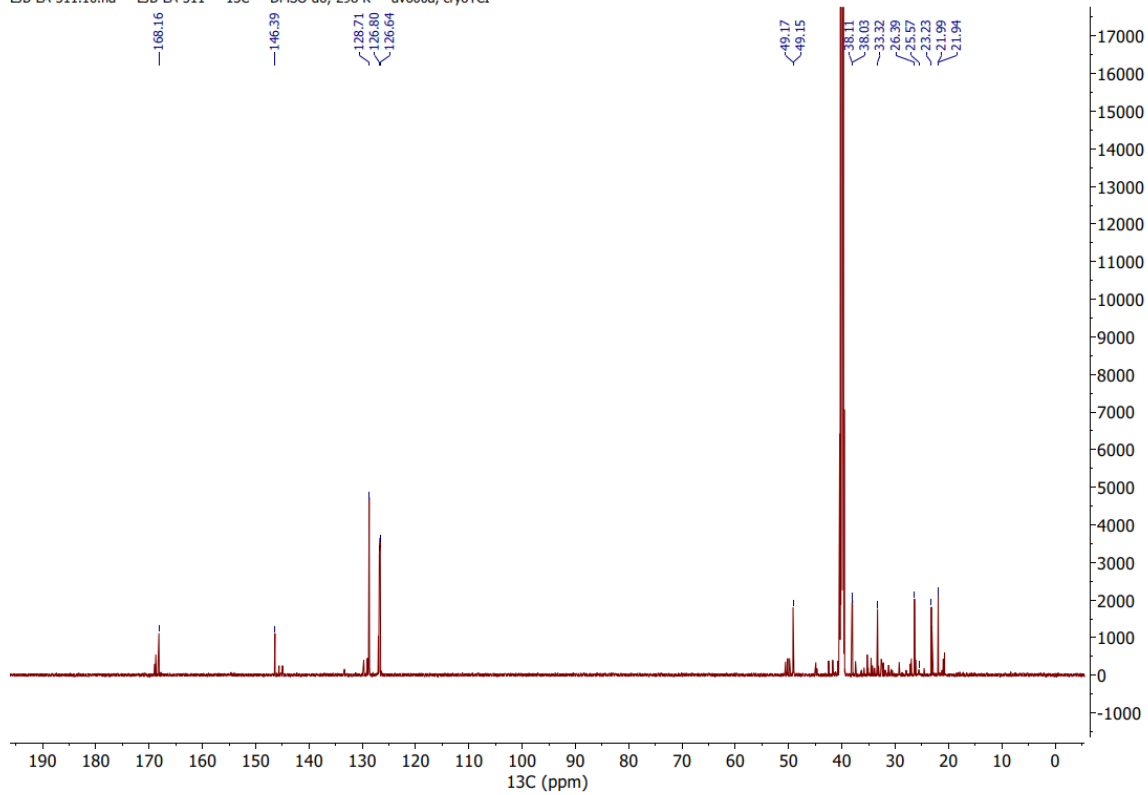
The other isomers in the mixture present too much signal overlap for reliable structure elucidation.

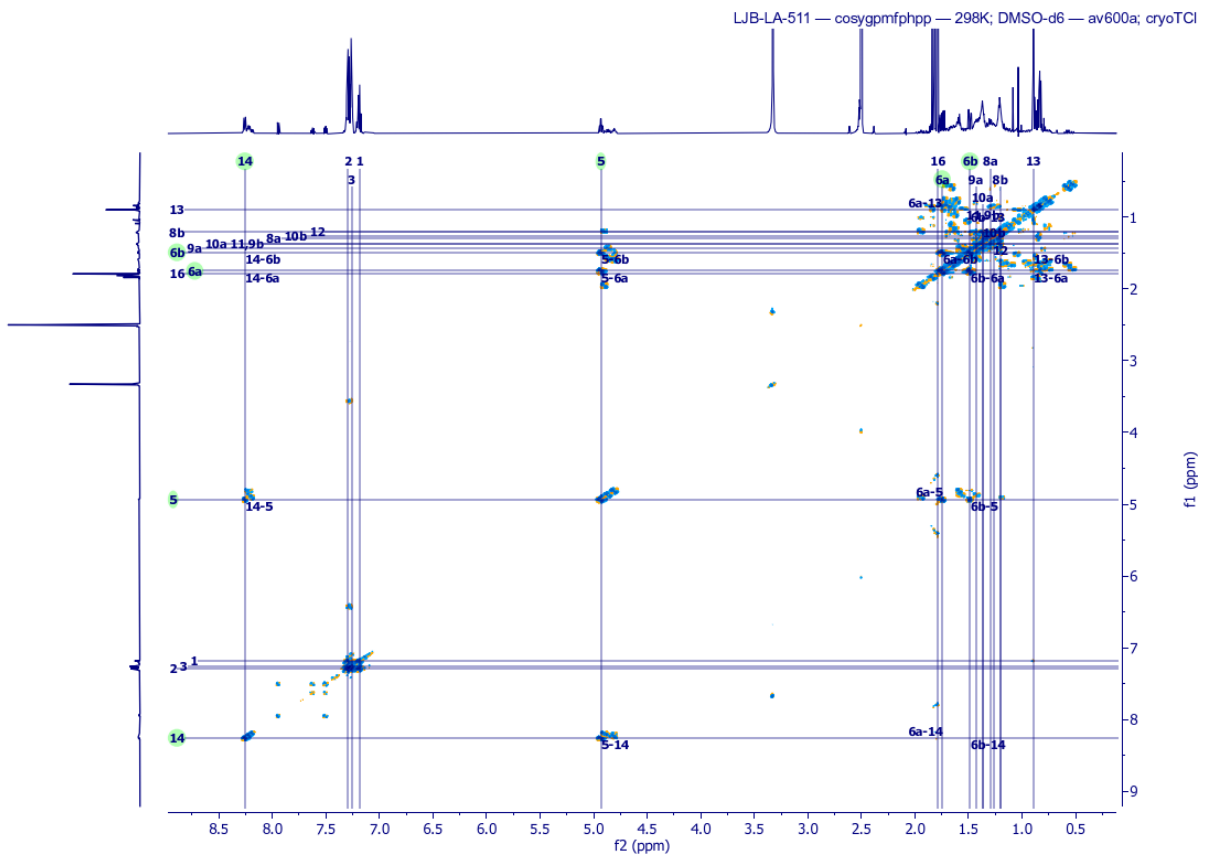
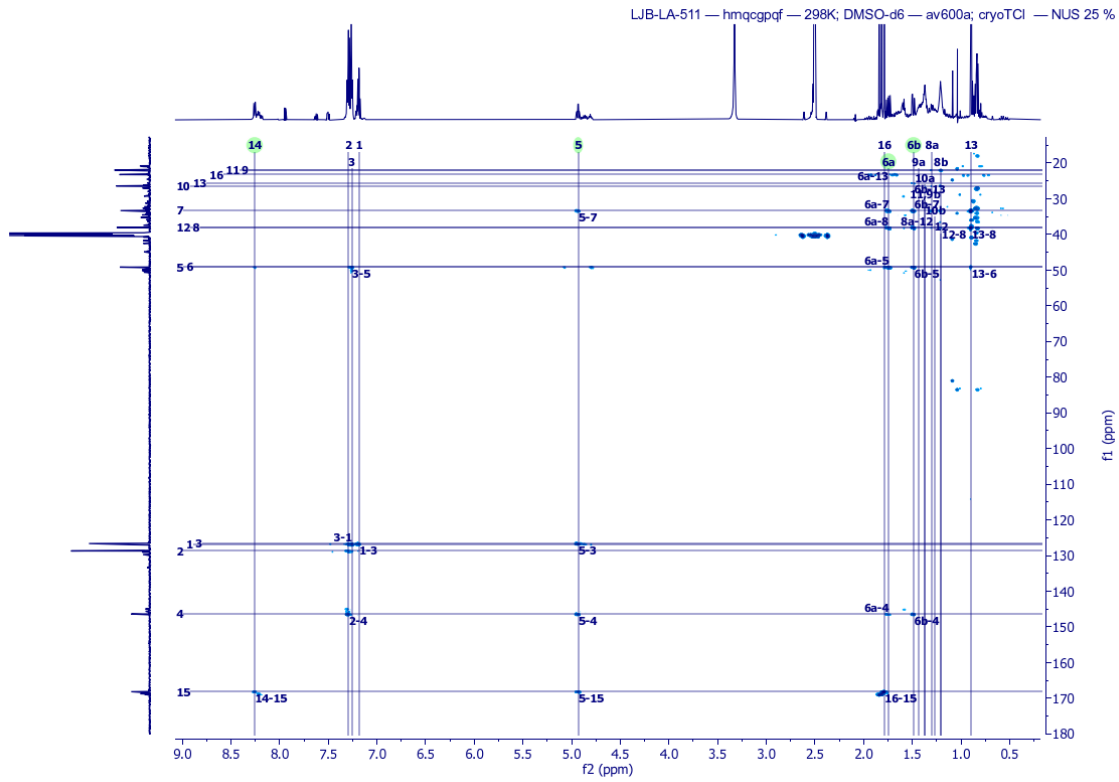
| Atom | δ (ppm) | J | HSQC | HMQC | COSY | NOESY | ¹⁵ N-HMBC |
|------|----------------|--------------------------|--------|--------------------|---------------|------------------|----------------------|
| C1 | 126.80 | | 1 | 3 | | | |
| H1 | 7.184 | m (overlapped) | 1 | 3 | | | |
| C2 | 128.71 | | 2 | | | | |
| H2 | 7.29 | m (overlapped) | 2 | 4 | | | |
| C3 | 126.64 | | 3 | 1, 5 | | | |
| H3 | 7.26 | m (overlapped) | 3 | 1, 5 | | 5, 6b, 14 | |
| C4 | 146.39 | | | 2, 5, 6a, 6b | | | |
| C5 | 49.17 | | 5 | 3, 6a, 6b, 16 | | | |
| H5 | 4.93 | d 3.5(6b), t 9.0(6a, 14) | 5 | 3, 4, 7, 15 | 6a, 6b, 14 | 3, 6b, 13, 14 | |
| C6 | 49.08 | | 6a, 6b | 13 | | | |
| H6a | 1.746 | d 9.1(5), d 14.4(6b) | 6 | 4, 5, 7, 8, 12, 13 | 5, 6b, 13, 14 | 14 | |
| H6b | 1.488 | d 3.5(5), d 14.4(6a) | 6 | 4, 5, 7, 8, 12, 13 | 5, 6a, 13, 14 | 3, 5 | |
| C7 | 33.32 | | | 5, 6a, 6b | | | |
| C8 | 38.03 | | 8a, 8b | 6a, 6b, 12, 13 | | | |
| H8a | 1.30 | m (overlapped) | 8 | 12 | | | |
| H8b | 1.21 | m (overlapped) | 8 | | | | |
| C9 | 21.94 | | 9 | | | | |
| H9 | 1.46-1.17 | m (overlapped) | 9 | | | | |
| C10 | 26.39 | | 10 | | | | |
| H10 | 1.46-1.17 | m (overlapped) | 10 | | | | |
| C11 | 21.99 | | 11 | | | | |
| H11 | 1.46-1.17 | m (overlapped) | 11 | | | | |
| C12 | 38.11 | | 12 | 6a, 6b, 8a, 13 | | | |
| H12 | 1.46-1.17 | m (overlapped) | 12 | 8 | | | |
| C13 | 25.56 | | 13 | 6a, 6b | | | |
| H13 | 0.896 | s | 13 | 6, 8, 12 | 6a, 6b | 5, 14 | |
| N14 | -246.5 | | | | | | 14, 16 |
| H14 | 8.257 | d 8.80(5) | | 15 | 5, 6a, 6b, 16 | 3, 5, 6a, 13, 16 | 14 |
| C15 | 168.16 | | | 5, 14, 16 | | | |
| C16 | 23.23 | | 16 | | | | |
| H16 | 1.789 | s | 16 | 5, 15 | 14 | 14 | 14 |

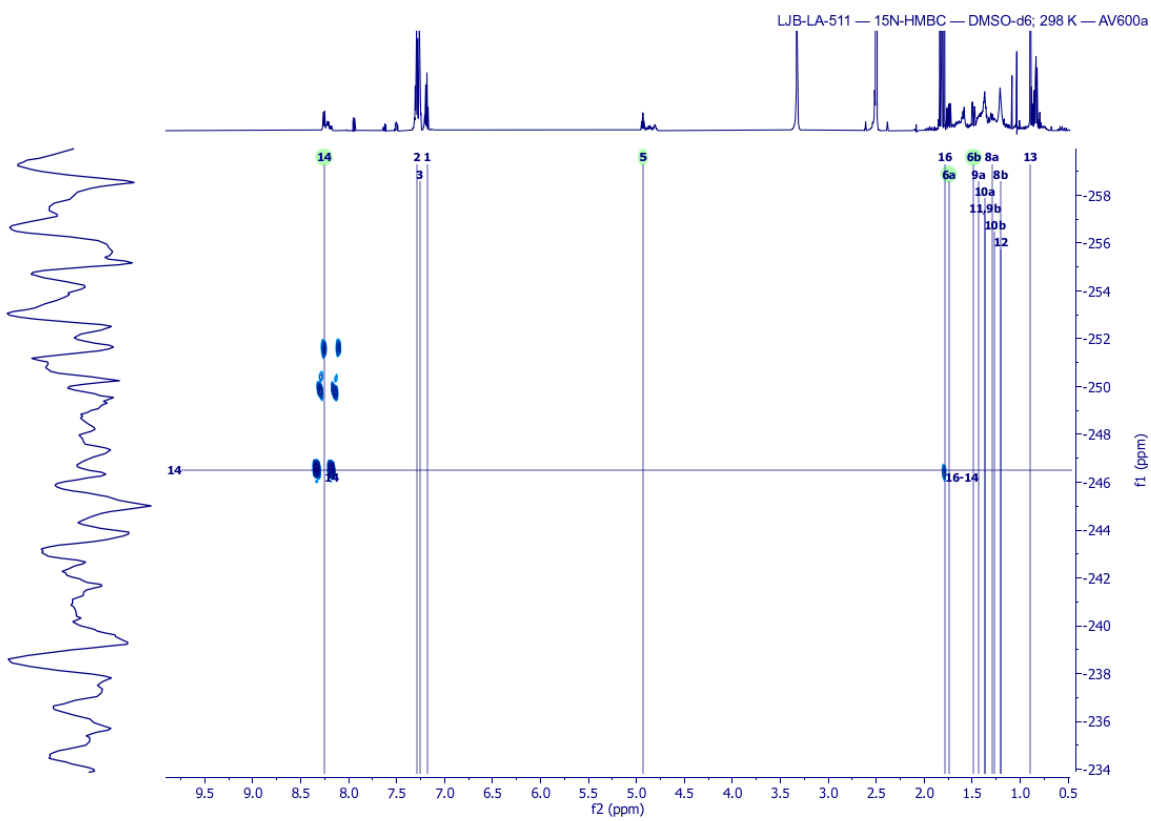
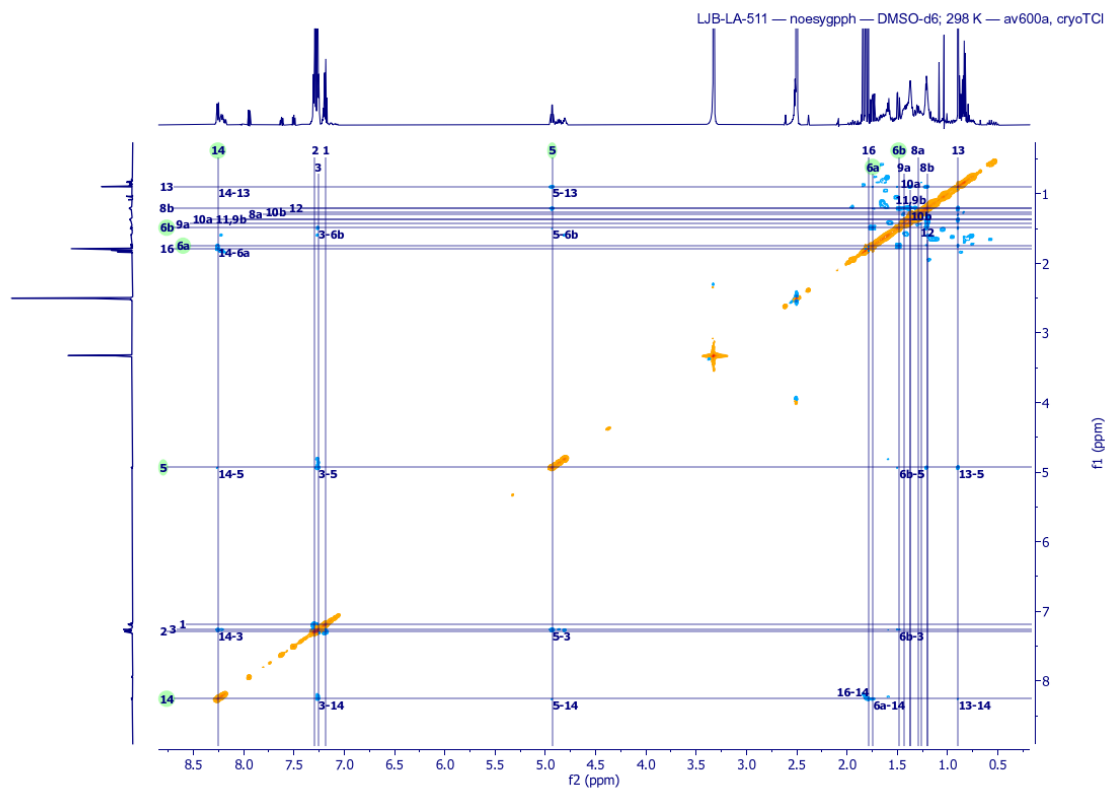
LJB-LA-511.13.fid — LJB-LA-511 — 1H — DMSO-d₆; 298 K; 2 mg — av600a, cryoTCl

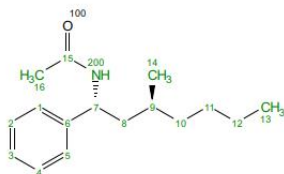


LJB-LA-511.10.fid — LJB-LA-511 — 13C — DMSO-d6; 298 K — av600a, cryoTCl



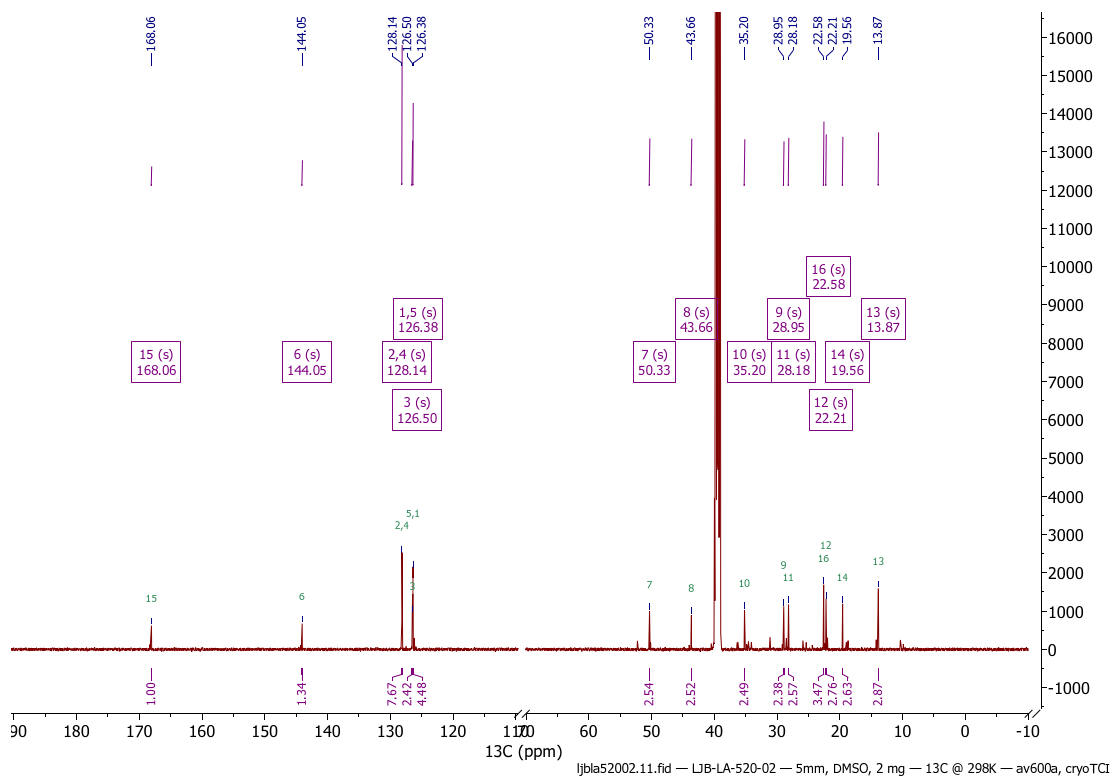
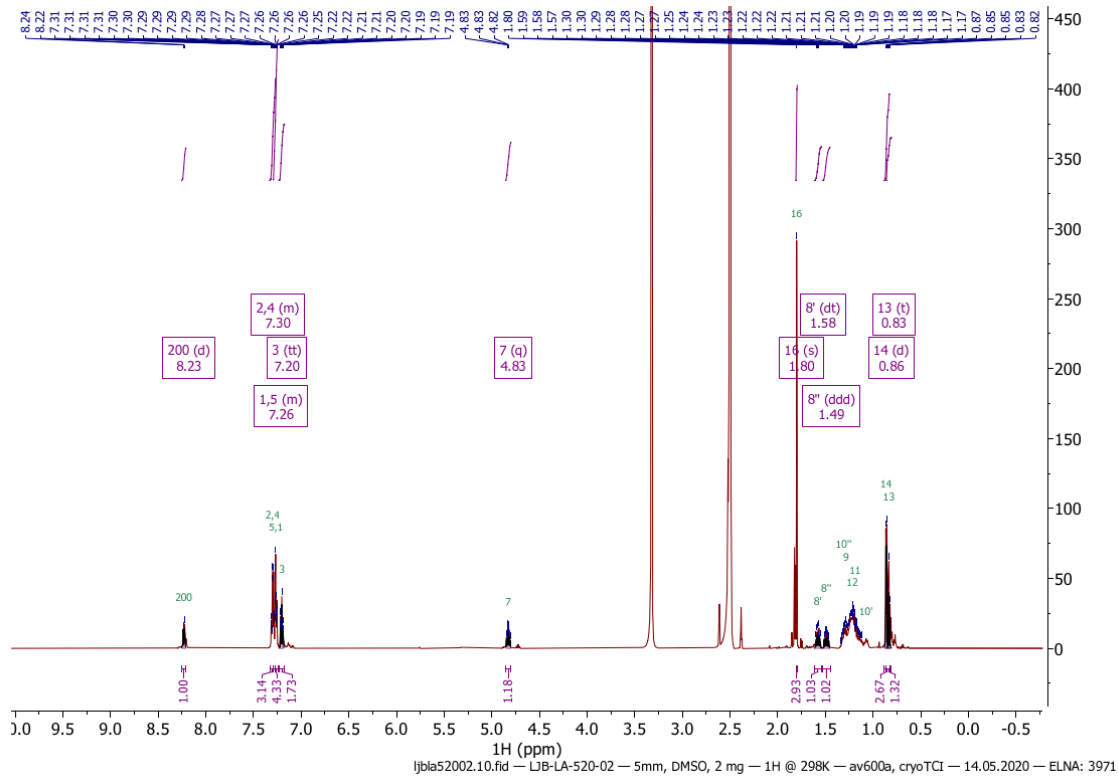


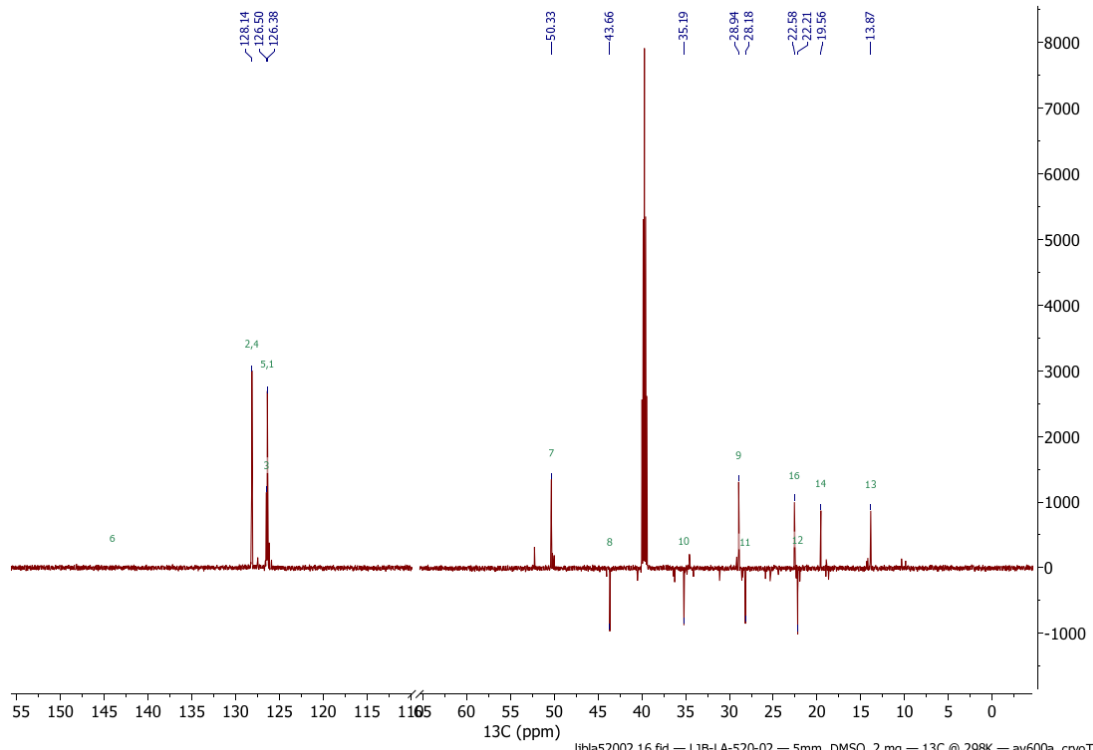




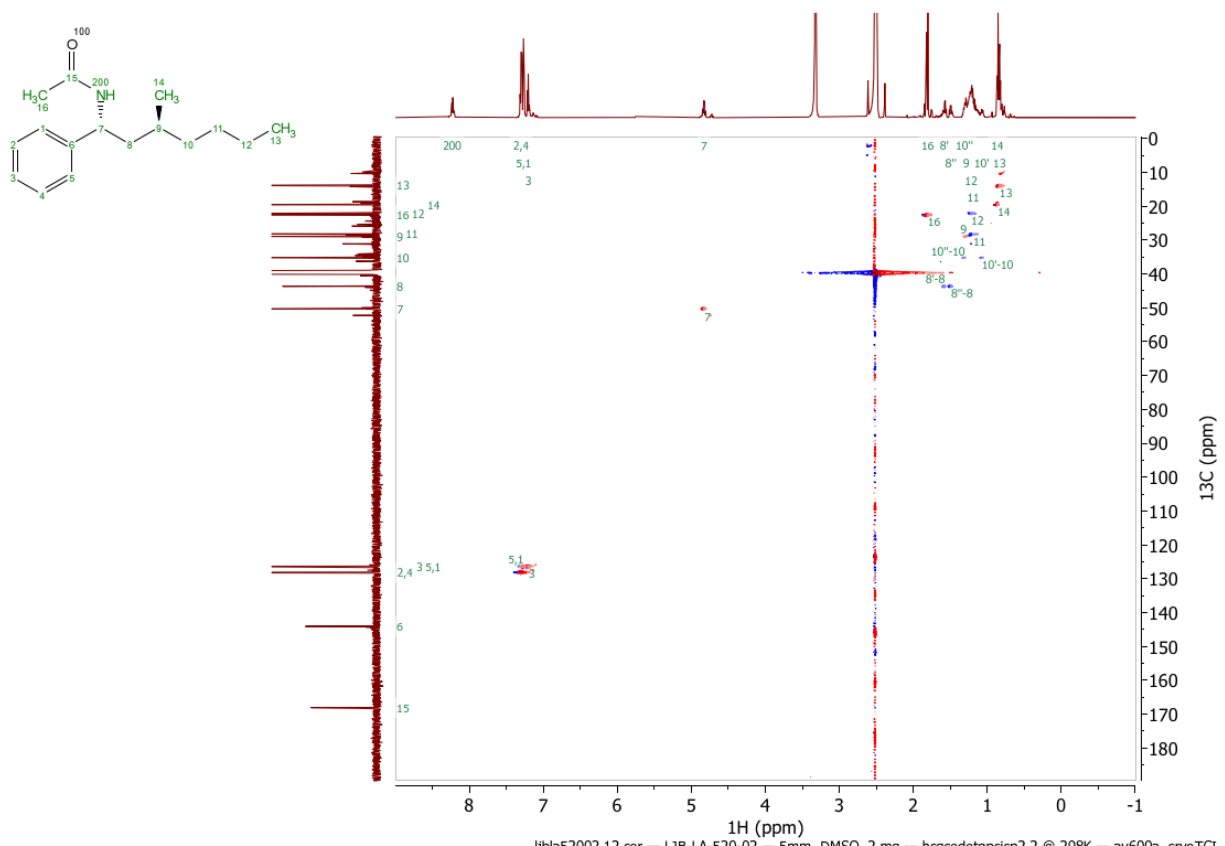
CH2-Groups are overlapping and can not be assigned with the Multiplet Manager individually.
 The measured 2D-Spectra support the suggested structure.
 Important HMBCs are: C9 - H7, C7 - H5 / H1, C15 - H7
 There are no NOEs between H200 - H14 and H14 and H16.
 Important NOEs are: H7 - H14 / H12 / H9 / H8, H200 - H1 / H7 / H16 / H8 / H9

| Atom | δ (ppm) | Min.,Max (ppm) | Predicted Shift | J | COEY | HSQC | HMBC | NOESY | Atom | δ (ppm) | Min.,Max (ppm) | Predicted Shift | J | COEY | HSQC | HMBC | NOESY |
|------|----------------|----------------|-----------------|---|-------------|------|-------------------|--|-------|----------------|----------------|-----------------|---------------------|------|----------|-------------|------------------------|
| 1 C | 126.38 | 126.35..126.41 | 127.29 | | | | | | 10 C | 35.2 | 35.14..35.25 | 36.98 | | | 10', 10' | 8', 8', 14 | |
| H | 7.26 | 7.24..7.29 | 7.34 | 1.50(3) | 2 | 1 | 3, 7 | 7, 8', 8', 9, 14, 16, 200 | H | 1.07 | 1.04..1.10 | 1.18, 1.30 | | 10' | 10 | 11 | 7, 9, 10', 11 |
| 2 C | 128.14 | 128.11..128.18 | 128.25 | | | 2 | 4 | | H' | 1.31 | 1.27..1.34 | 1.18, 1.30 | | 10' | 10 | | 7, 10', 11 |
| H | 7.3 | 7.27..7.33 | 7.32 | 7.20(3) | 1, 3 | 2 | 4, 6 | 7 | 11 C | 28.18 | 28.13..28.22 | 29.47 | | | 11 | 10', 12, 13 | |
| 3 C | 126.5 | 126.44..126.56 | 127.66 | | | 3 | 1, 5 | | H'E | 1.18 | 1.12..1.25 | 1.22, 1.28 | | | 11 | 13 | 7, 10', 10', 12 |
| H | 7.2 | 7.17..7.23 | 7.26 | 7.20(2), 1.50(1), 7.20(4), 1.50(5), 2.00(?) | 2, 4 | 3 | 1, 5 | | 12 C | 22.21 | 22.16..22.25 | 23.09 | | | 12 | 13 | |
| 4 C | 128.14 | 128.11..128.18 | 128.25 | | | 4 | 2 | | H'E | 1.22 | 1.16..1.28 | 1.24, 1.26 | | 13 | 12 | 11 | 8', 11, 13 |
| H | 7.3 | 7.27..7.33 | 7.32 | 7.20(3) | 3, 5 | 4 | 2, 6 | 7 | 13 C | 13.87 | 13.83..13.90 | 14.08 | | | 13 | 11 | |
| 5 C | 126.38 | 126.35..126.41 | 127.29 | | | 5 | 3, 7 | | H'B | 0.83 | 0.81..0.86 | 0.89 | 6.97(?), 6.97(?) | 12 | 13 | 11, 12 | 12 |
| H | 7.26 | 7.24..7.29 | 7.34 | 1.50(3) | 4 | 5 | 3, 7 | 7, 8', 8', 9, 14, 16, 200 | 14 C | 19.56 | 19.52..19.61 | 18.96 | | | 14 | 8', 8' | |
| 6 C | 144.05 | 144.01..144.09 | 143.24 | | | | 2, 4, 7, 8', 8' | | H'B | 0.86 | 0.83..0.88 | 0.91 | 6.62(?) | 9 | 14 | 8, 9, 10 | 1, 5, 7, 8', 8', 9 |
| 7 C | 50.33 | 50.27..50.38 | 51.39 | | | 7 | 1, 5, 8', 8', 200 | | 15 C | 168.06 | 168.03..168.10 | 169.5 | | | | 7, 16, 200 | |
| H | 4.83 | 4.79..4.86 | 4.96 | 8.80(200), 8.52(8'), 7.02(?) | 8', 8', 200 | 7 | 1, 5, 6, 8, 9, 15 | 1, 2, 4, 5, 8', 8', 9, 10', 10', 11, 14, 200 | 16 C | 22.58 | 22.53..22.63 | 23.11 | | | 16 | | |
| 8 C | 43.66 | 43.59..43.72 | 38.39 | | | | 8', 8' | 7, 14, 200 | H'B | 1.8 | 1.79..1.81 | 1.85 | | | 16 | 15 | 1, 5, 200 |
| H' | 1.58 | 1.54..1.62 | 1.64, 1.88 | 13.63(?), 6.86(?), 6.86(?) | 7, 8', 9 | 8 | 6, 7, 9, 10, 14 | 1, 5, 7, 8', 9, 12, 14, 200 | 100 O | | | | | | | | |
| H' | 1.49 | 1.45..1.53 | 1.64, 1.88 | 13.42(?), 6.60(?) | 7, 8', 9 | 8 | 6, 7, 9, 10, 14 | 1, 5, 7, 8', 9, 14, 200 | 200 N | | | | | | | | |
| 9 C | 28.95 | 28.90..28.99 | 29.77 | | | 9 | 7, 8', 8', 14 | | H | 8.23 | 8.21..8.25 | 6.14 | 8.80(?) | 7 | | 7, 8, 15 | 1, 5, 7, 8', 8', 9, 16 |
| H | 1.28 | 1.25..1.31 | 1.39 | | | | 8', 8', 14 | 1, 5, 7, 8', 8', 10', 14, 200 | | | | | | | | | |

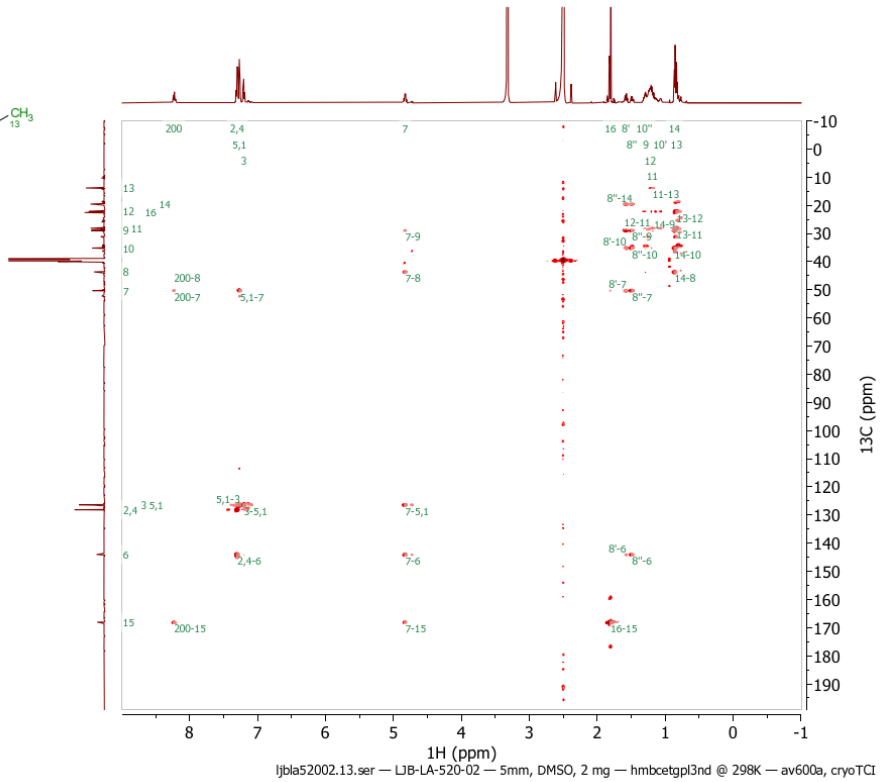
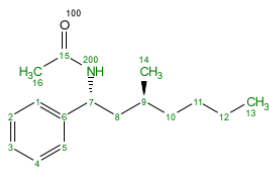


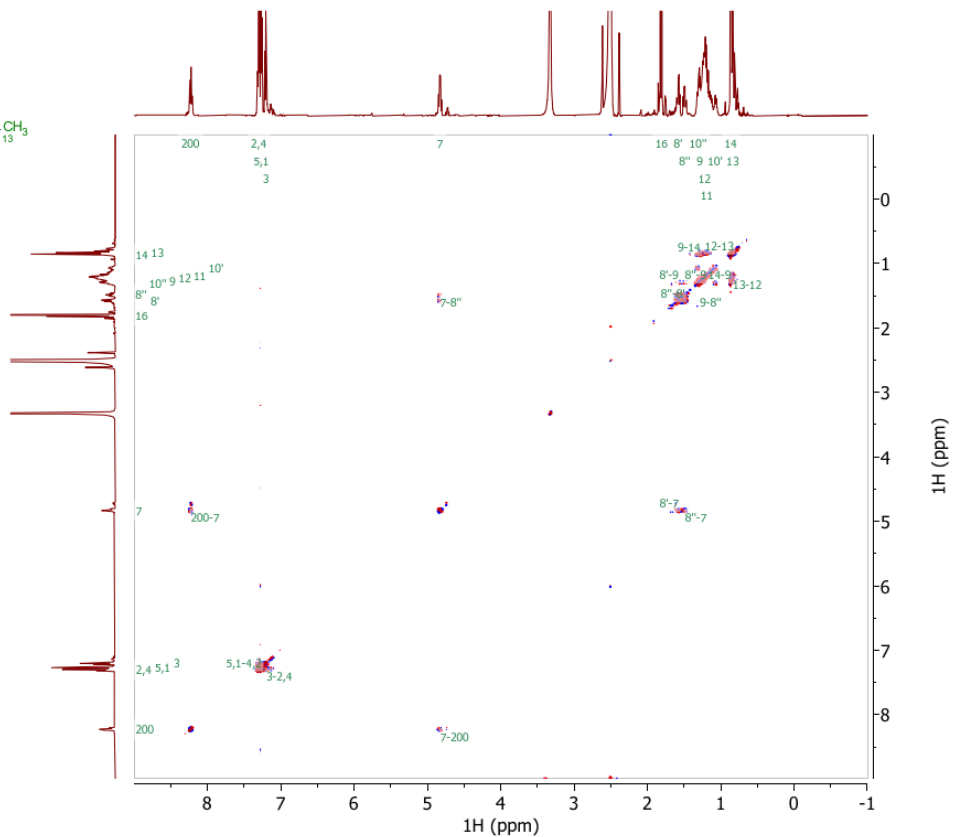
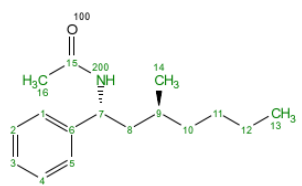


ljbla52002.16.fid — LJB-LA-520-02 — 5mm, DMSO, 2 mg — 13C @ 298K — av600a, cryoTCl

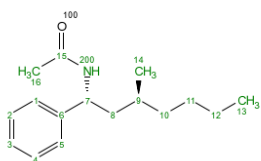


ljbla52002.12.ser — LJB-LA-520-02 — 5mm, DMSO, 2 mg — hsqcdetgpcisp2.2 @ 298K — av600a, cryoTCl

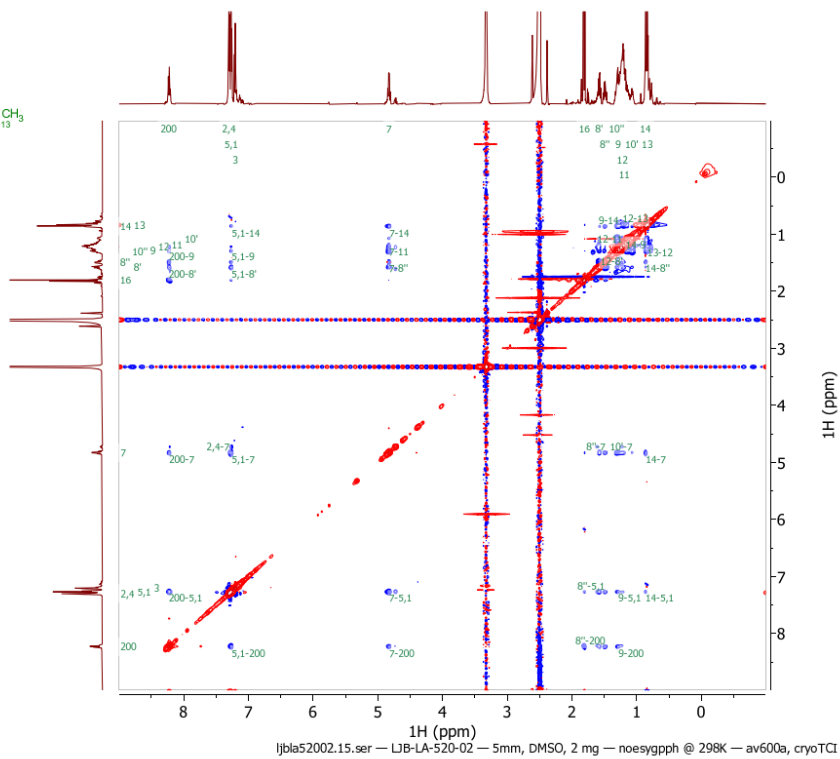




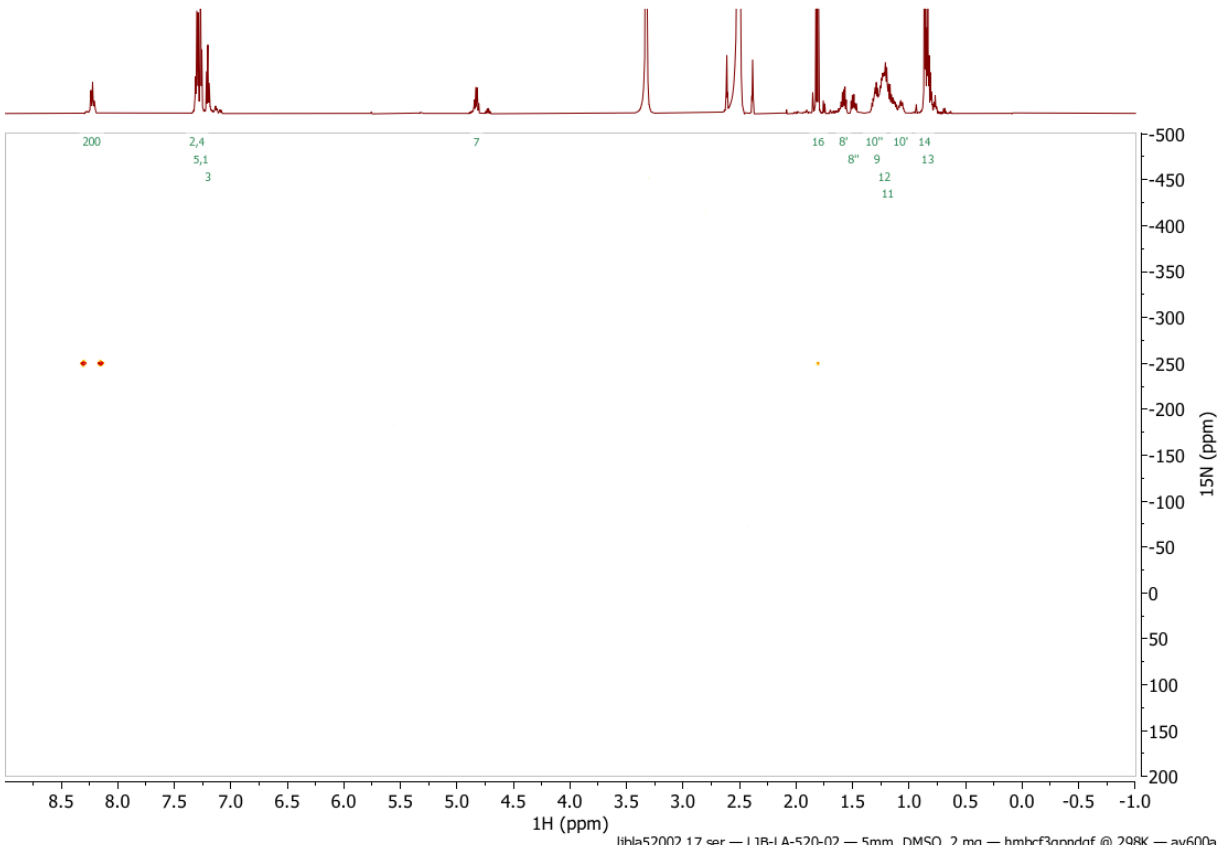
ljbla52002.14.ser — LJB-LA-520-02 — 5mm, DMSO, 2 mg — cosygpmfphpp @ 298K — av600a, cryoTCI

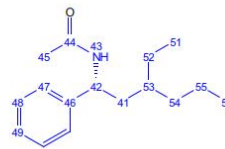
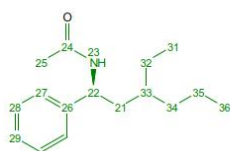
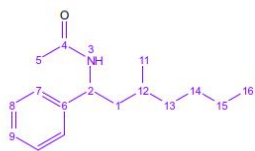


See NOE-arrows with mouse over atoms.



ljbla52002.15.ser — LJB-LA-520-02 — 5mm, DMSO, 2 mg — noesygpph @ 298K — av600a, cryoTCI



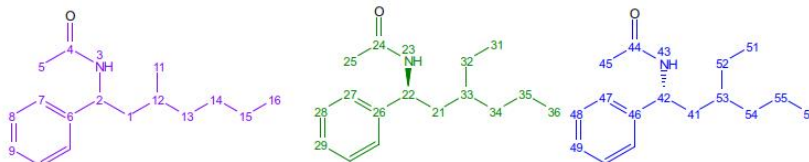


¹H NMR (600 MHz, DMSO) δ 8.20 (dd, $J = 8.7, 3.7$ Hz, 1H, 3, 43, 23), 7.32 – 7.23 (m, 4H, 7, 8, 47, 48, 27, 28), 7.19 (ddt, $J = 8.7, 5.4, 1.6$ Hz, 1H, 9, 49, 29), 4.89 – 4.78 (m, 1H, 2, 42, 22), 1.83 – 1.78 (m, 2H, 5, 45, 25), 1.66 (ddd, $J = 13.3, 10.5, 4.4$ Hz, 0H, 1'), 1.56 (dp, $J = 14.0, 4.3$ Hz, 0H, 41', 21'), 1.47 (dtd, $J = 13.6, 6.3, 3.7$ Hz, 0H, 41', 21'), 1.41 (d, $J = 5.5$ Hz, 0H, 12), 1.34 – 1.25 (m, 1H, 1'), 1.29 – 1.15 (m, 7H, 13', 14', 15', 52', 53', 54', 55', 32', 33', 34', 35'), 1.14 – 1.07 (m, 0H, 13'), 0.91 – 0.82 (m, 3H, 11, 16, 36), 0.82 – 0.77 (m, 1H, 51, 56), 0.75 (t, $J = 7.2$ Hz, 1H, 31).

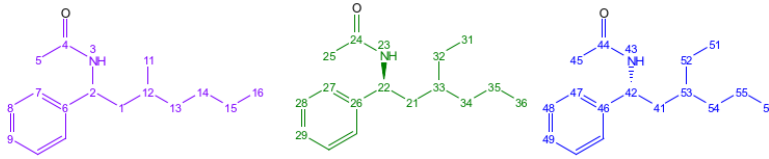
¹³C NMR (151 MHz, DMSO) δ 168.83 (4), 168.73 (24), 168.69 (44), 145.12 (6), 144.86 (26), 144.81 (46), 128.66 (8, 48, 28), 126.92 (9, 49, 29), 126.74 (7, 47, 27), 50.73 (42), 50.67 (22), 50.53 (2), 44.54 (1), 41.02 (41), 40.99 (21), 36.90 (13), 35.37 (34), 35.09 (53), 35.02 (33), 34.63 (54), 29.68 (12), 29.02 (14), 25.88 (52), 24.93 (32), 23.10 (5, 45, 25), 22.89 (15), 19.51 (35), 19.47 (11), 19.21 (55), 14.83 (36), 14.72 (56), 14.46 (16), 10.87 (51), 10.41 (31).

Notes:

- This is a mixture of products. The signals are heavily overlapped in ¹H, but thankfully many signals can be resolved in ¹³C.
- Therefore, the assignment is mainly based on predictions from the suggested products...
- The C-C bond with hexane was formed at position 2 in the major product and at position 3 in the other two minor products which are diastereomers of one another.
- We cannot assign which diastereomer is which.
- The ratios is approx:3:2:1:5
- The HMBC, COSY and NOESY spectra were not assigned

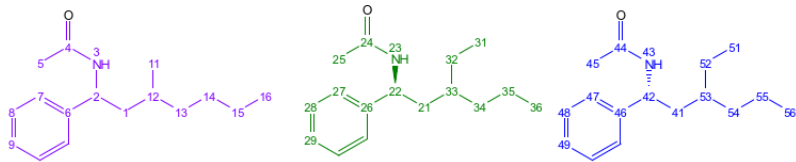
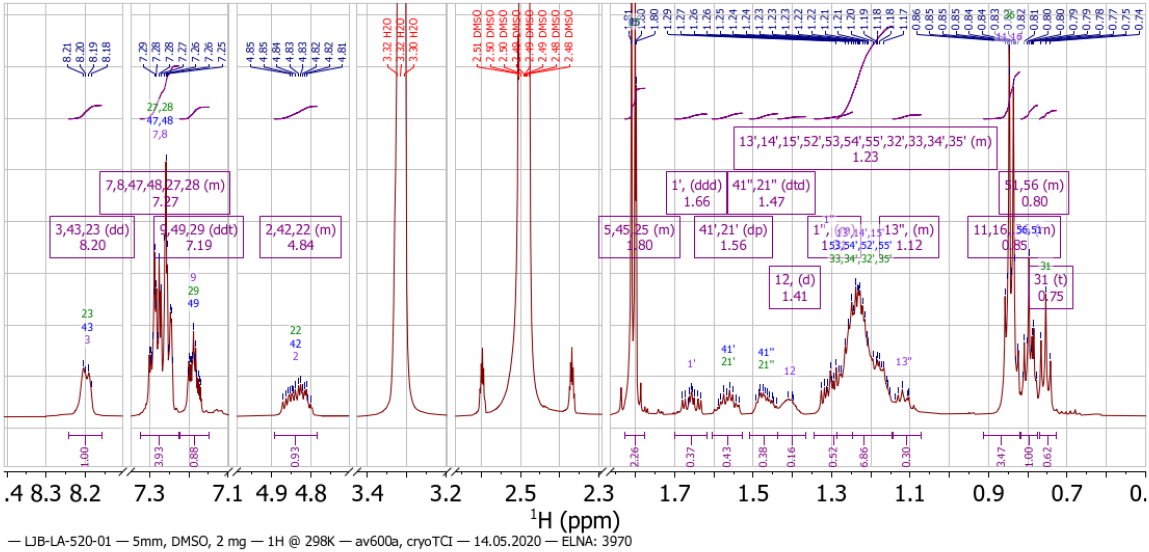


| Atom | δ (ppm) | Predicted Shift | HSQC | Atom | δ (ppm) | Predicted Shift | HSQC | Atom | δ (ppm) | Predicted Shift | HSQC |
|------|----------------|-----------------|-----------|------|----------------|-----------------|-----------|------|----------------|-----------------|-----------|
| 1 C | 44.54 | 41.31 | 1', 1'' | 21 C | 40.99 | 39 | 21', 21'' | 41 C | 41.02 | 39 | 41', 41'' |
| H' | 1.66 | 1.73, 1.77 | 1 | H' | 1.56 | 1.78, 1.81 | 21 | H' | 1.56 | 1.78, 1.81 | 41 |
| H'' | 1.31 | 1.73, 1.77 | 1 | H'' | 1.47 | 1.78, 1.81 | 21 | H'' | 1.47 | 1.78, 1.81 | 41 |
| 2 C | 50.53 | 52.3 | 2 | 22 C | 50.67 | 52.14 | 22 | 42 C | 50.73 | 52.15 | 42 |
| H | 4.84 | 4.85 | 2 | H | 4.84 | 4.82 | 22 | H | 4.84 | 4.81 | 42 |
| 3 N | -250.7 | | | 23 N | -250.7 | | | 43 N | -250.7 | | |
| H | 8.2 | 6.8 | | H | 8.2 | 6.64 | | H | 8.2 | 6.64 | |
| 4 C | 168.83 | 169.82 | | 24 C | 168.73 | 169.82 | | 44 C | 168.69 | 169.82 | |
| 5 C | 23.1 | 23.28 | 5 | 25 C | 23.1 | 23.28 | | 45 C | 23.1 | 23.28 | 45 |
| H3 | 1.8 | 1.88 | 5 | H3 | 1.8 | 1.88 | | H3 | 1.8 | 1.88 | 45 |
| 6 C | 145.12 | 143.37 | | 26 C | 144.86 | 143.36 | | 46 C | 144.81 | 143.36 | |
| 7 C | 126.74 | 125.86 | 7 | 27 C | 126.74 | 125.86 | 27 | 47 C | 126.74 | 125.86 | 47 |
| H | 7.27 | 7.3 | 7 | H | 7.27 | 7.3 | 27 | H | 7.27 | 7.3 | 47 |
| 8 C | 128.66 | 128.33 | 8 | 28 C | 128.66 | 128.33 | 28 | 48 C | 128.66 | 128.33 | 48 |
| H | 7.27 | 7.32 | 8 | H | 7.27 | 7.32 | 28 | H | 7.27 | 7.32 | 48 |
| 9 C | 126.92 | 127.47 | 9 | 29 C | 126.92 | 127.47 | 29 | 49 C | 126.92 | 127.47 | 49 |
| H | 7.19 | 7.27 | 9 | H | 7.19 | 7.27 | 29 | H | 7.19 | 7.27 | 49 |
| 11 C | 19.47 | 20.55 | 11 | 31 C | 10.41 | 11.1 | 31 | 51 C | 10.87 | 11.1 | 51 |
| H3 | 0.85 | 0.9 | 11 | H3 | 0.75 | 0.86 | 31 | H3 | 0.8 | 0.86 | 51 |
| 12 C | 29.68 | 30.39 | 12 | 32 C | 24.93 | 27.31 | 32', 32'' | 52 C | 25.88 | 27.31 | 52', 52'' |
| H | 1.41 | 1.6 | 12 | H' | 1.23 | 1.28, 1.31 | 32 | H' | 1.23 | 1.28, 1.31 | 52 |
| 13 C | 36.9 | 36.64 | 13', 13'' | H'' | 1.28 | 1.31 | 32 | H'' | 1.28 | 1.31 | 52 |
| H' | 1.23 | 1.23, 1.25 | 13 | 33 C | 35.02 | 36.87 | 33 | 53 C | 35.09 | 36.87 | 53 |
| H'' | 1.12 | 1.23, 1.25 | 13 | H | 1.23 | 1.6 | 33 | H | 1.23 | 1.6 | 53 |
| 14 C | 29.02 | 29.4 | 14', 14'' | 34 C | 35.37 | 36.46 | 34', 34'' | 54 C | 34.63 | 36.46 | 54', 54'' |
| H' | 1.23 | 1.25, 1.27 | 14 | H' | 1.23 | 1.23, 1.25 | 34 | H' | 1.23 | 1.23, 1.25 | 54 |
| H'' | | 1.25, 1.27 | 14 | H'' | | 1.23, 1.25 | 34 | H'' | | 1.23, 1.25 | 54 |
| 15 C | 22.89 | 23 | 15', 15'' | 35 C | 19.51 | 20.05 | 35', 35'' | 55 C | 19.21 | 20.05 | 55', 55'' |
| H' | 1.23 | 1.26, 1.27 | 15 | H' | 1.23 | 1.28, 1.29 | 35 | H' | 1.23 | 1.28, 1.29 | 55 |
| H'' | | 1.26, 1.27 | 15 | H'' | | 1.28, 1.29 | 35 | H'' | | 1.28, 1.29 | 55 |
| 16 C | 14.46 | 14.11 | 16 | 36 C | 14.83 | 14.48 | 36 | 56 C | 14.72 | 14.48 | 56 |
| H3 | 0.85 | 0.89 | 16 | H3 | 0.85 | 0.89 | 36 | H3 | 0.8 | 0.89 | 56 |



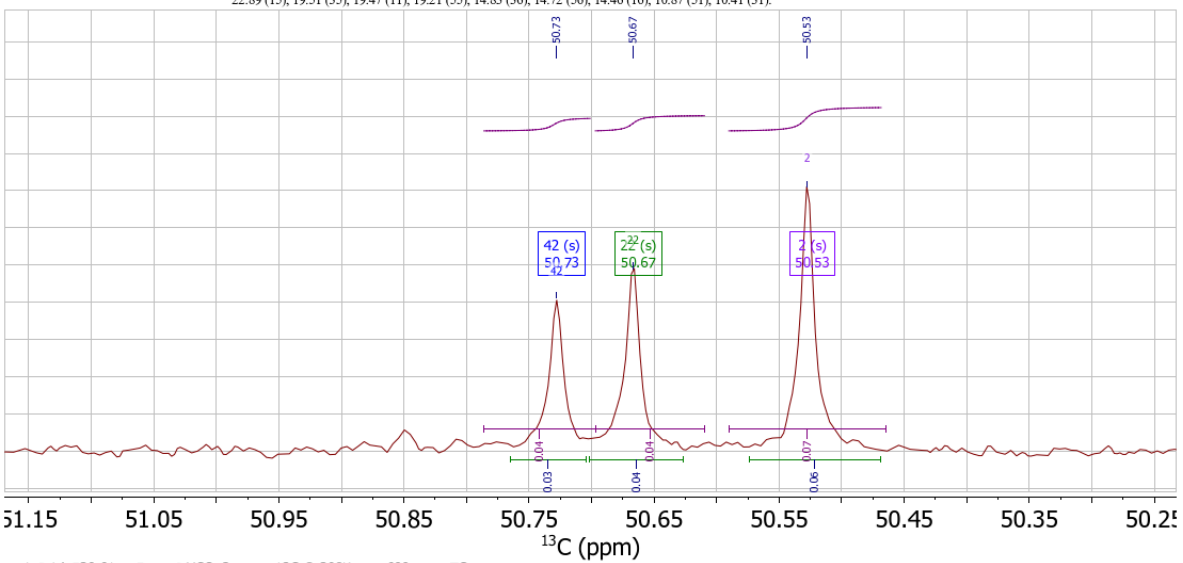
1D ¹H

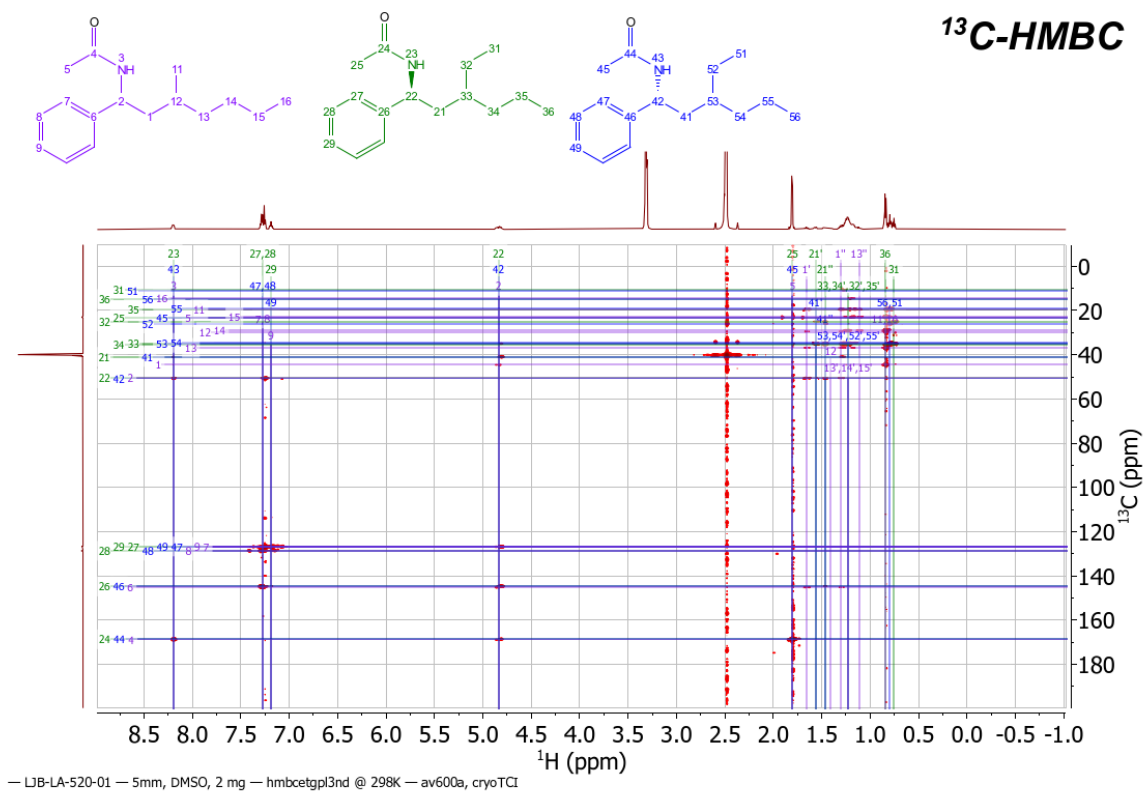
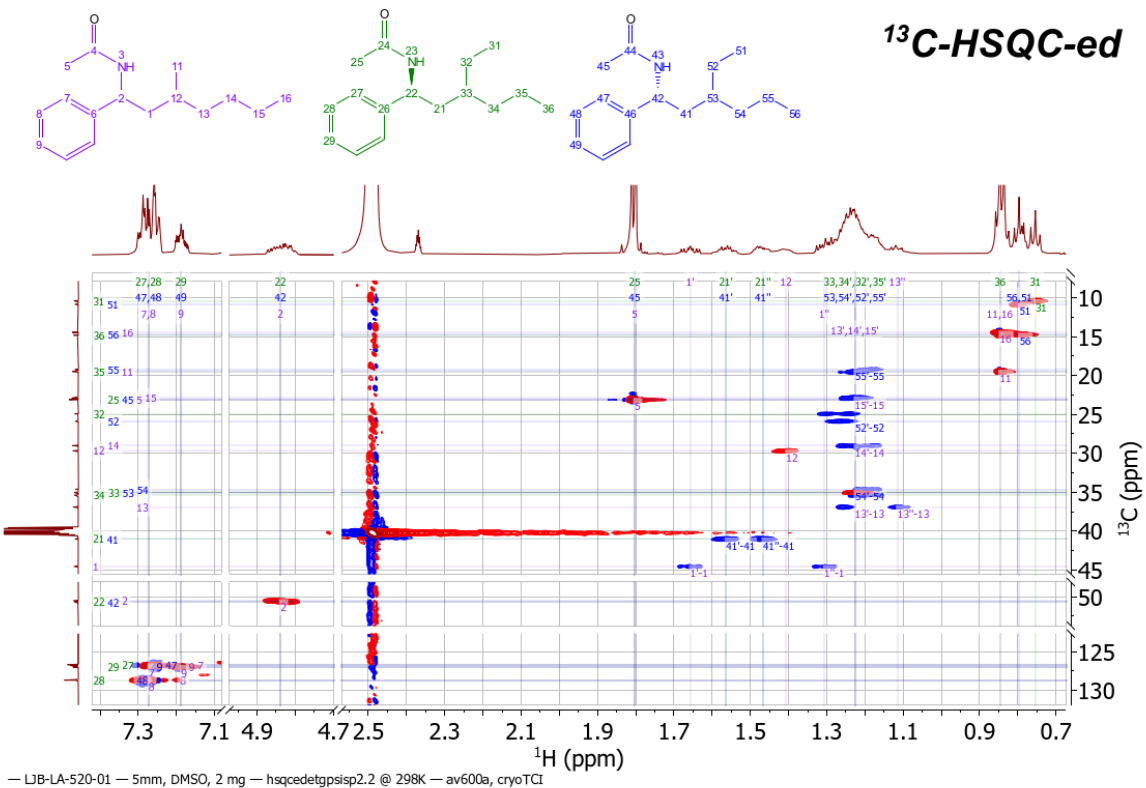
¹H NMR (600 MHz, DMSO) δ 8.20 (dd, *J* = 8.7, 3.7 Hz, 1H, 3, 43, 23), 7.32 – 7.23 (m, 4H, 7, 8, 47, 48, 27, 28), 7.19 (ddt, *J* = 8.7, 5.4, 1.6 Hz, 1H, 9, 49, 29), 4.89 – 4.78 (m, 1H, 2, 42, 22), 1.83 – 1.78 (m, 2H, 5, 45, 25), 1.66 (ddd, *J* = 13.3, 10.5, 4.4 Hz, 0H, 1'), 1.56 (dp, *J* = 14.0, 4.3 Hz, 0H, 41', 21'), 1.47 (dtd, *J* = 13.6, 6.3, 3.7 Hz, 0H, 41', 21'), 1.41 (d, *J* = 5.5 Hz, 0H, 12), 1.34 – 1.25 (m, 1H, 1'), 1.29 – 1.15 (m, 7H, 13', 14', 15', 52', 53, 54', 55', 32', 33, 34', 35'), 1.14 – 1.07 (m, 0H, 13'), 0.91 – 0.82 (m, 3H, 11, 16, 36), 0.82 – 0.77 (m, 1H, 51, 56), 0.75 (t, *J* = 7.2 Hz, 1H, 31).



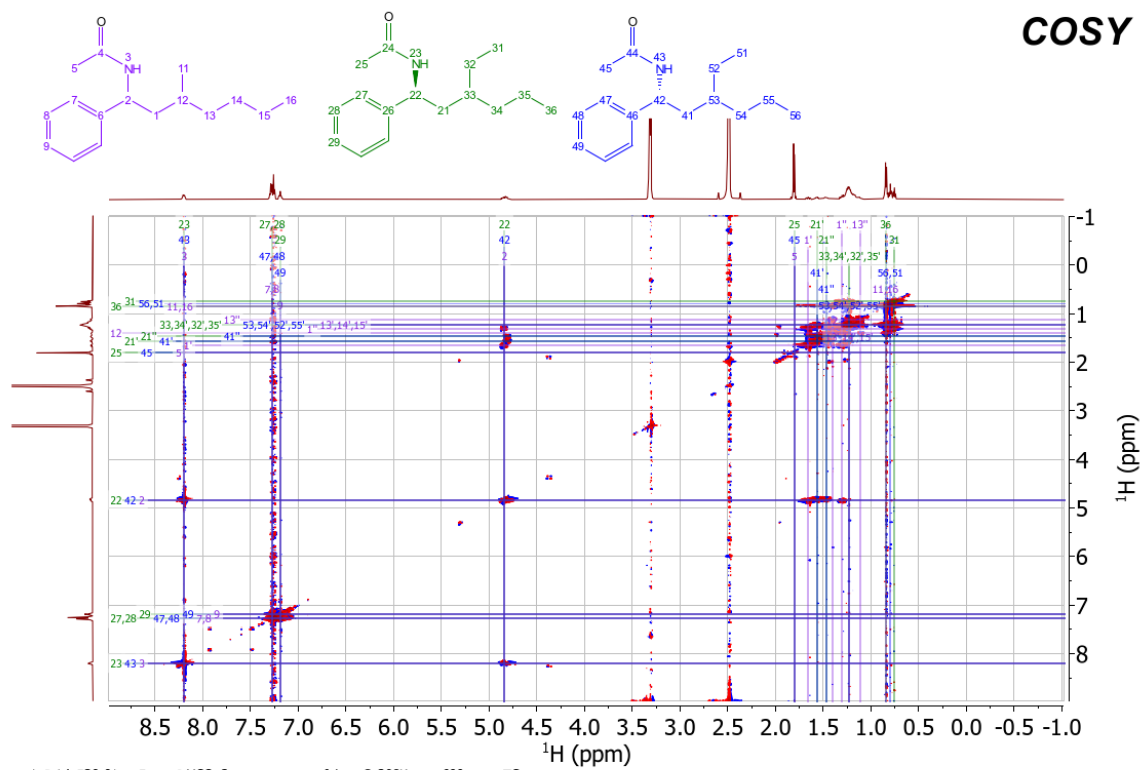
1D ¹³C{¹H}

¹³C NMR (151 MHz, DMSO) δ 168.83 (4), 168.73 (24), 168.69 (44), 145.12 (6), 144.86 (26), 144.81 (46), 128.66 (8, 48, 28), 126.92 (9, 49, 29), 126.74 (7, 47, 27), 50.73 (42), 50.67 (22), 50.53 (2), 44.54 (1), 41.02 (41), 40.99 (21), 36.90 (13), 35.37 (34), 35.09 (53), 35.02 (33), 34.63 (54), 29.68 (12), 29.02 (14), 25.88 (52), 24.93 (32), 23.10 (5, 45, 25), 22.89 (15), 19.51 (35), 19.47 (11), 19.21 (55), 14.83 (36), 14.72 (56), 14.46 (16), 10.87 (51), 10.41 (31).

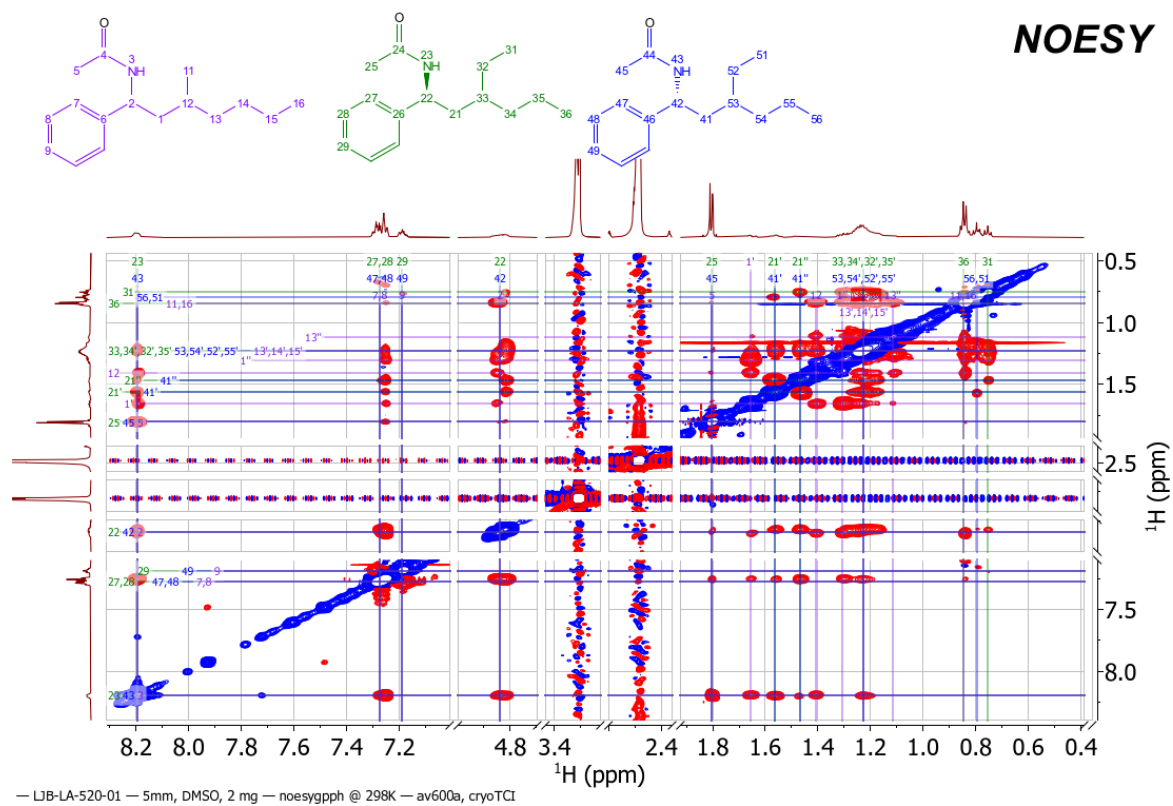




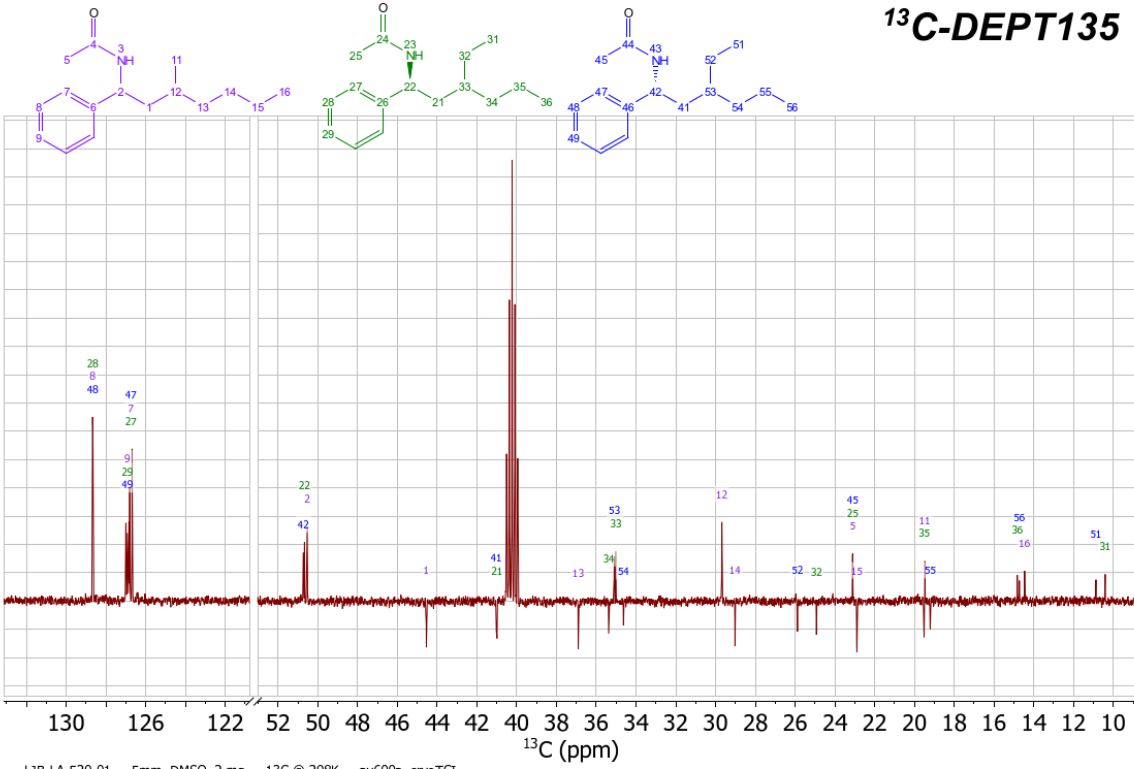
COSY



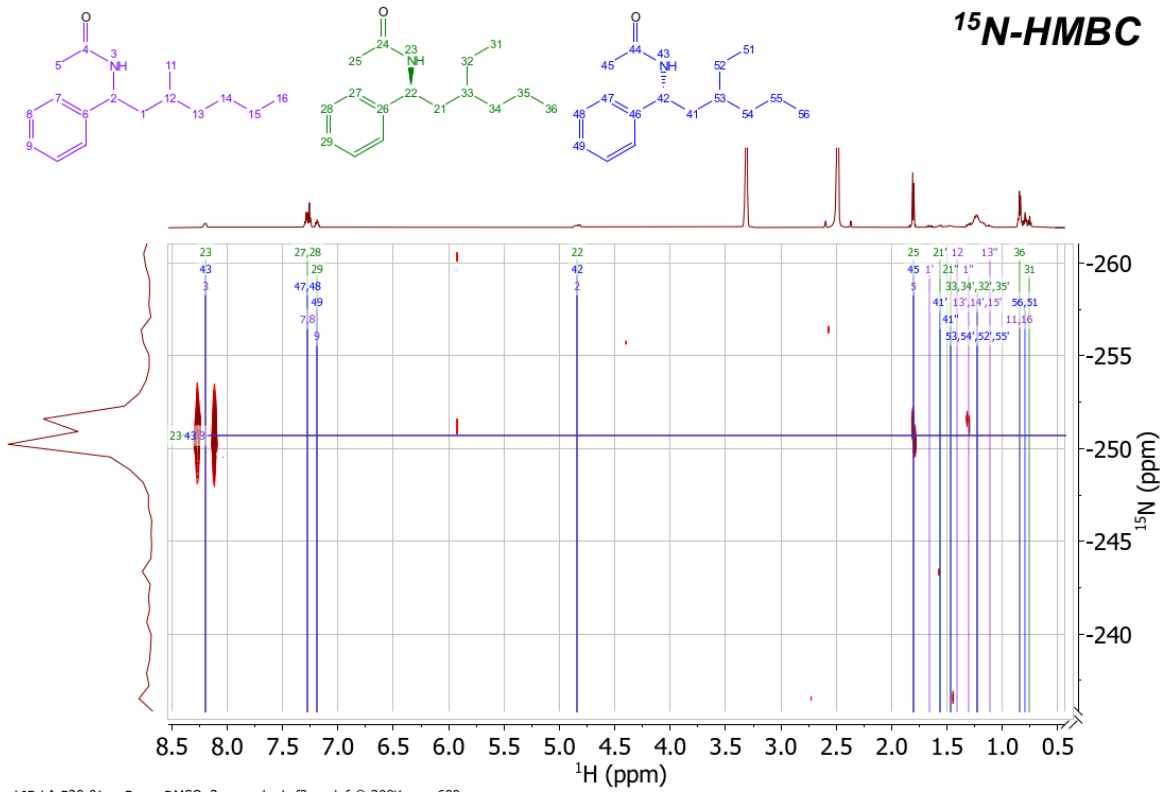
NOESY



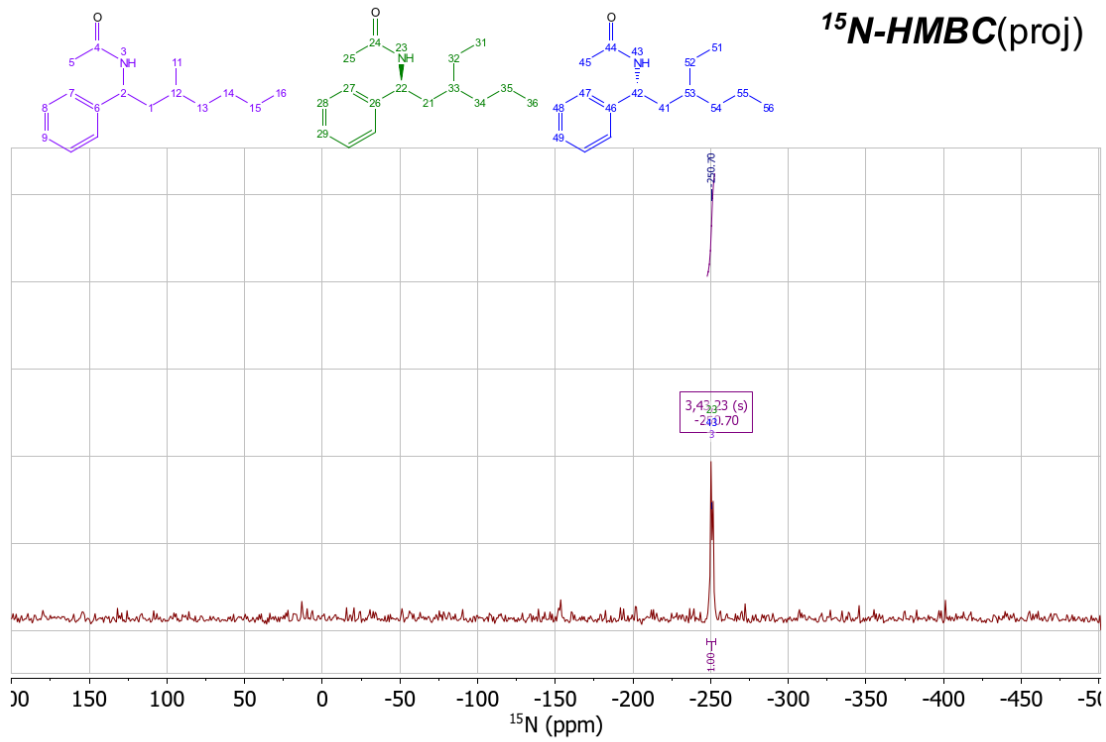
¹³C-DEPT135



¹⁵N-HMBC



¹⁵N-HMBC(proj)



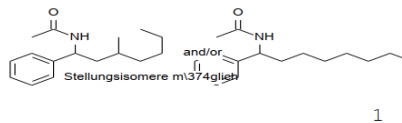
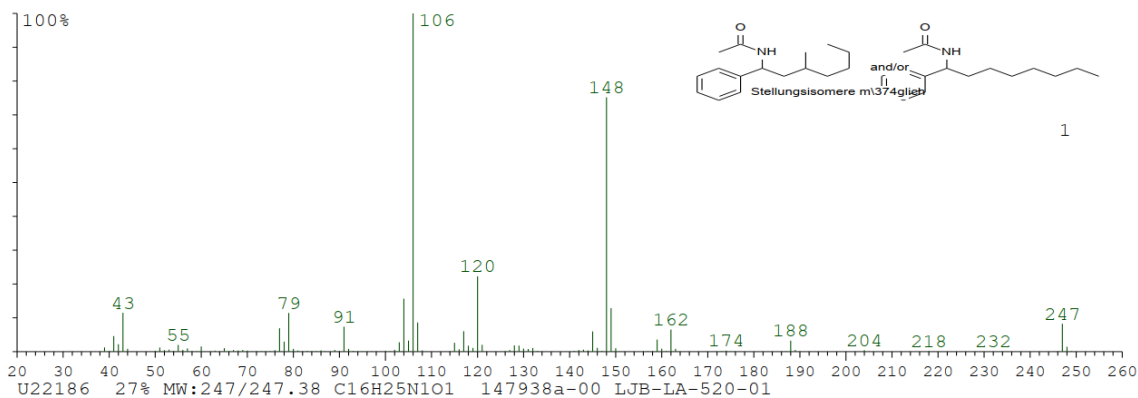
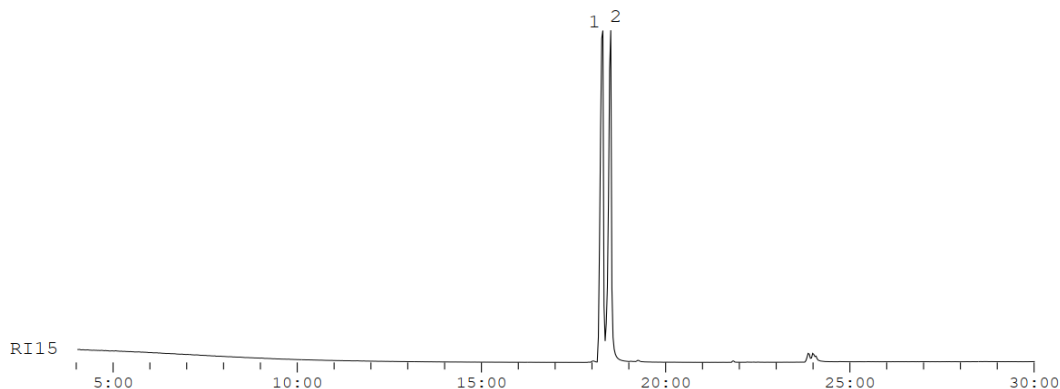
| No. | MW. | Comment |
|-----|-----|---|
| 1 | 247 | Your proposed structures are possible. Ref.-spectrum: U22186:147938a-00 LJB-LA-520-01 |
| 2 | 247 | Your proposed structures are possible. Equal to Peak 1 Ref.-spectrum: U22187:147938a-00 LJB-LA-520-01 |

Other measurements will follow (HRMS)

6.07.2020
File: 147938a-00.raw
Analyse: LJB-LA-520-01
KMA: Liu, Sensheng

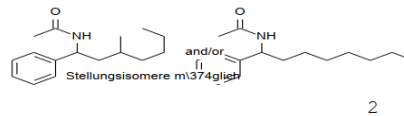
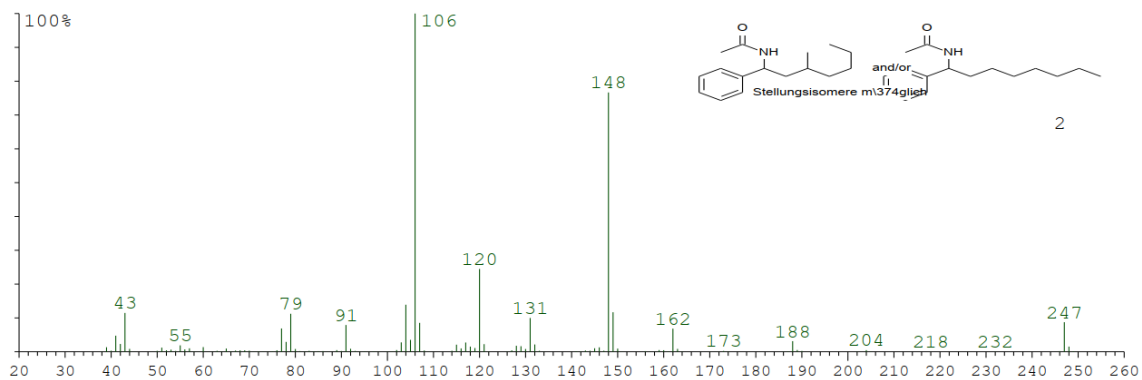
Messung: GC-MS
Ionisierung: GC-EI
Spektrometer: ISQ Series
Säule: MS 84 TG-5 SILMS
Länge: 30
Temp.: 35-5-285-5
GC-Nr.: -
ELNA-Nr.: 25947

Auswerter: Haupt (2243)



| U22186 27% MW:247/247.38 C16H25N1O1 147938a-00 LJB-LA-520-01 | | | | | | | | | | | | | |
|--|-------|----|-------|-----|-------|-----|--------|-----|-------|-----|------|-----|------|
| 38 | 0.06 | 63 | 0.30 | 84 | 0.13 | 105 | 3.27 | 130 | 0.91 | 155 | 0.03 | 200 | 0.02 |
| 39 | 1.24 | 64 | 0.11 | 85 | 0.18 | 106 | 100.00 | 131 | 0.78 | 156 | 0.16 | 202 | 0.07 |
| 40 | 0.25 | 65 | 1.03 | 86 | 0.37 | 107 | 8.62 | 132 | 1.09 | 157 | 0.12 | 204 | 0.45 |
| 41 | 4.60 | 66 | 0.15 | 87 | 0.11 | 108 | 0.39 | 133 | 0.23 | 158 | 0.14 | 205 | 0.07 |
| 42 | 2.20 | 67 | 0.48 | 88 | 0.03 | 109 | 0.04 | 134 | 0.07 | 159 | 3.57 | 218 | 0.15 |
| 43 | 11.49 | 68 | 0.35 | 89 | 0.52 | 110 | 0.03 | 139 | 0.02 | 160 | 0.88 | 219 | 0.02 |
| 44 | 0.83 | 69 | 0.49 | 90 | 0.23 | 112 | 0.02 | 140 | 0.02 | 161 | 0.18 | 232 | 0.02 |
| 45 | 0.04 | 70 | 0.31 | 91 | 7.37 | 113 | 0.02 | 141 | 0.25 | 162 | 6.53 | 246 | 0.13 |
| 50 | 0.27 | 71 | 0.08 | 92 | 0.82 | 114 | 0.17 | 142 | 0.49 | 163 | 0.84 | 247 | 8.25 |
| 51 | 1.20 | 72 | 0.15 | 93 | 0.23 | 115 | 2.63 | 143 | 0.59 | 164 | 0.06 | 248 | 1.44 |
| 52 | 0.43 | 73 | 0.11 | 94 | 0.11 | 116 | 0.71 | 144 | 0.37 | 170 | 0.05 | 249 | 0.12 |
| 53 | 0.60 | 74 | 0.08 | 95 | 0.07 | 117 | 6.04 | 145 | 5.98 | 172 | 0.03 | | |
| 54 | 0.26 | 75 | 0.14 | 96 | 0.06 | 118 | 1.76 | 146 | 1.12 | 173 | 0.14 | | |
| 55 | 1.99 | 76 | 0.45 | 97 | 0.10 | 119 | 1.10 | 147 | 0.26 | 174 | 0.31 | | |
| 56 | 0.53 | 77 | 6.91 | 98 | 0.10 | 120 | 22.27 | 148 | 75.19 | 175 | 0.04 | | |
| 57 | 0.96 | 78 | 2.96 | 99 | 0.03 | 121 | 2.02 | 149 | 12.86 | 176 | 0.11 | | |
| 58 | 0.17 | 79 | 11.43 | 100 | 0.27 | 122 | 0.09 | 150 | 1.03 | 186 | 0.05 | | |
| 59 | 0.06 | 80 | 0.82 | 101 | 0.10 | 126 | 0.12 | 151 | 0.07 | 187 | 0.12 | | |
| 60 | 1.53 | 81 | 0.34 | 102 | 0.55 | 127 | 0.48 | 152 | 0.03 | 188 | 3.24 | | |
| 61 | 0.04 | 82 | 0.14 | 103 | 2.77 | 128 | 1.85 | 153 | 0.02 | 189 | 0.49 | | |
| 62 | 0.05 | 83 | 0.25 | 104 | 15.65 | 129 | 1.78 | 154 | 0.03 | 190 | 0.06 | | |

U22186% MW:247/247.38 C16H25N1O1 147938a-00 LJB-LA-520-01 lim: 0.02%



U22187 27% MW:247/247.38 C16H25N1O1 147938a-00 LJB-LA-520-01

| | | | | | | | | | | | | | |
|----|-------|----|-------|-----|-------|-----|--------|-----|-------|-----|------|-----|------|
| 38 | 0.06 | 62 | 0.05 | 83 | 0.28 | 105 | 3.51 | 130 | 0.81 | 156 | 0.18 | 202 | 0.08 |
| 39 | 1.33 | 63 | 0.29 | 84 | 0.14 | 106 | 100.00 | 131 | 9.98 | 157 | 0.09 | 203 | 0.02 |
| 40 | 0.31 | 64 | 0.10 | 85 | 0.18 | 107 | 8.54 | 132 | 2.15 | 158 | 0.06 | 204 | 0.47 |
| 41 | 4.75 | 65 | 0.95 | 86 | 0.19 | 108 | 0.37 | 133 | 0.29 | 159 | 0.50 | 205 | 0.07 |
| 42 | 2.30 | 66 | 0.13 | 87 | 0.06 | 109 | 0.05 | 134 | 0.07 | 160 | 0.45 | 218 | 0.03 |
| 43 | 11.47 | 67 | 0.35 | 88 | 0.03 | 110 | 0.03 | 139 | 0.02 | 161 | 0.15 | 232 | 0.06 |
| 44 | 0.84 | 68 | 0.41 | 89 | 0.48 | 112 | 0.03 | 141 | 0.16 | 162 | 6.83 | 245 | 0.02 |
| 45 | 0.04 | 69 | 0.42 | 90 | 0.22 | 113 | 0.02 | 142 | 0.12 | 163 | 0.84 | 246 | 0.19 |
| 49 | 0.03 | 70 | 0.32 | 91 | 7.91 | 114 | 0.29 | 143 | 0.35 | 164 | 0.06 | 247 | 8.74 |
| 50 | 0.27 | 71 | 0.07 | 92 | 0.87 | 115 | 2.09 | 144 | 0.29 | 170 | 0.04 | 248 | 1.52 |
| 51 | 1.22 | 72 | 0.15 | 93 | 0.24 | 116 | 0.99 | 145 | 1.03 | 171 | 0.02 | 249 | 0.14 |
| 52 | 0.42 | 73 | 0.08 | 94 | 0.11 | 117 | 2.73 | 146 | 1.30 | 172 | 0.04 | | |
| 53 | 0.62 | 74 | 0.08 | 95 | 0.13 | 118 | 1.52 | 147 | 0.31 | 173 | 0.24 | | |
| 54 | 0.24 | 75 | 0.14 | 96 | 0.07 | 119 | 1.10 | 148 | 76.66 | 174 | 0.11 | | |
| 55 | 1.89 | 76 | 0.44 | 97 | 0.12 | 120 | 24.47 | 149 | 11.68 | 176 | 0.02 | | |
| 56 | 0.70 | 77 | 6.88 | 98 | 0.05 | 121 | 2.25 | 150 | 0.93 | 186 | 0.07 | | |
| 57 | 1.01 | 78 | 2.91 | 100 | 0.08 | 122 | 0.09 | 151 | 0.06 | 187 | 0.14 | | |
| 58 | 0.11 | 79 | 11.23 | 101 | 0.09 | 126 | 0.09 | 152 | 0.02 | 188 | 3.13 | | |
| 59 | 0.05 | 80 | 0.81 | 102 | 0.50 | 127 | 0.42 | 153 | 0.02 | 189 | 0.52 | | |
| 60 | 1.37 | 81 | 0.20 | 103 | 2.77 | 128 | 1.77 | 154 | 0.02 | 190 | 0.23 | | |
| 61 | 0.04 | 82 | 0.12 | 104 | 13.90 | 129 | 1.64 | 155 | 0.02 | 191 | 0.03 | | |

U22187% MW:247/247.38 C16H25N1O1 147938a-00 LJB-LA-520-01 lim: 0.02%

Peak 1

Mass to be matched (m/z): 247.193180 Charge: 1

Mass Tolerance: ±0.050000

Restriction of atom numbers:

C H N O

1-100 1-100 1-2 1-2

Number of calculated Formulas: 5

| Formula | Diff. (ppm) | theor. m/z |
|---------------|-------------|------------|
| C16 H25 N1 O1 | -0.47 | 247.193064 |
| C15 H23 N2 O1 | -51.35 | 247.180488 |
| C15 H21 N1 O2 | -147.66 | 247.156679 |
| C13 H31 N2 O2 | 181.32 | 247.238002 |
| C14 H19 N2 O2 | -198.54 | 247.144102 |

Suggestion:

C16H25N1O1 MW: 247

7.07.2020

File: 147938b-00.raw

Analyse: LJB-LA-520-01

KMA: Liu, Sensheng

Messung: GC-MS
 Ionisierung: GC-EI
 Spektrometer: Q Exactive GC Orbitrap
 Säule: MS 75 ZB-5HT 30+5
 Länge: 30+5
 Temp.: 35-10-285-5
 GC-Nr.: -
 ELNA-Nr.: 25947

Auswerter: Haupt (2243)

Peak 2

Mass to be matched (m/z): 247.192960 Charge: 1

Mass Tolerance: ±0.050000

Restriction of atom numbers:

C H N O

1-100 1-100 1-2 1-2

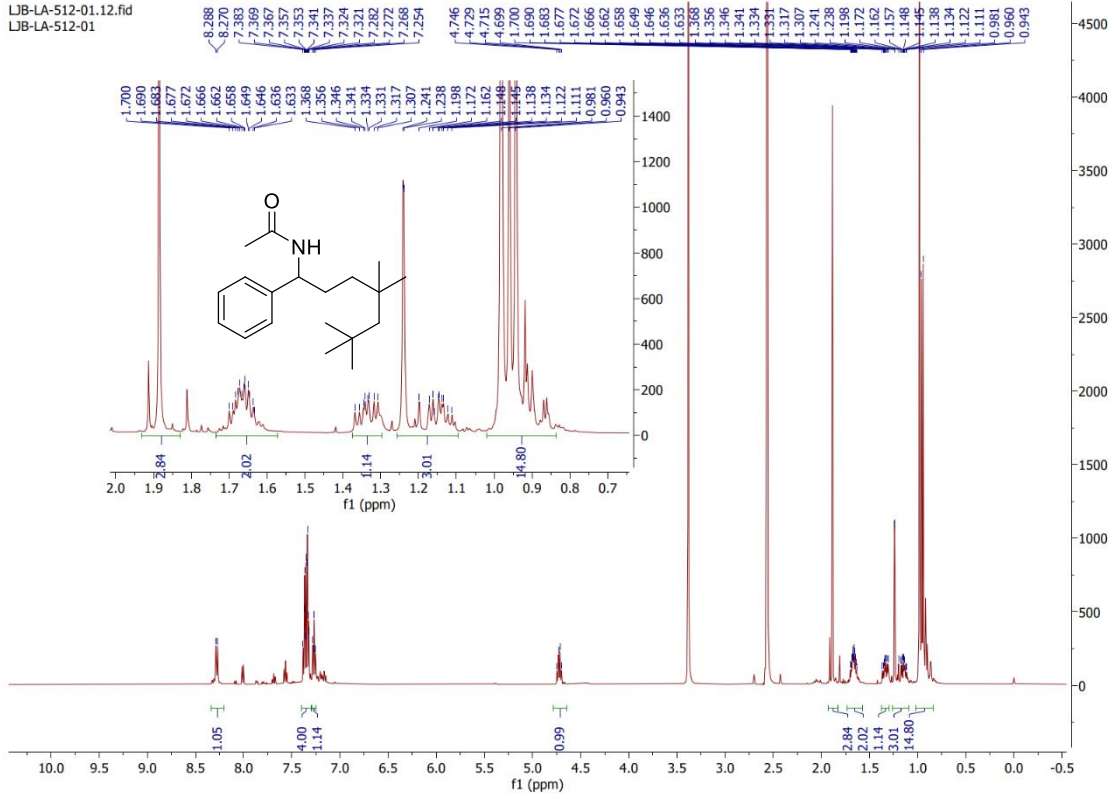
Number of calculated Formulas: 5

| Formula | Diff. (ppm) | theor. m/z |
|---------------|-------------|------------|
| C16 H25 N1 O1 | 0.42 | 247.193063 |
| C15 H23 N2 O1 | -50.46 | 247.180487 |
| C15 H21 N1 O2 | -146.77 | 247.156679 |
| C13 H31 N2 O2 | 182.22 | 247.238003 |
| C14 H19 N2 O2 | -197.65 | 247.144102 |

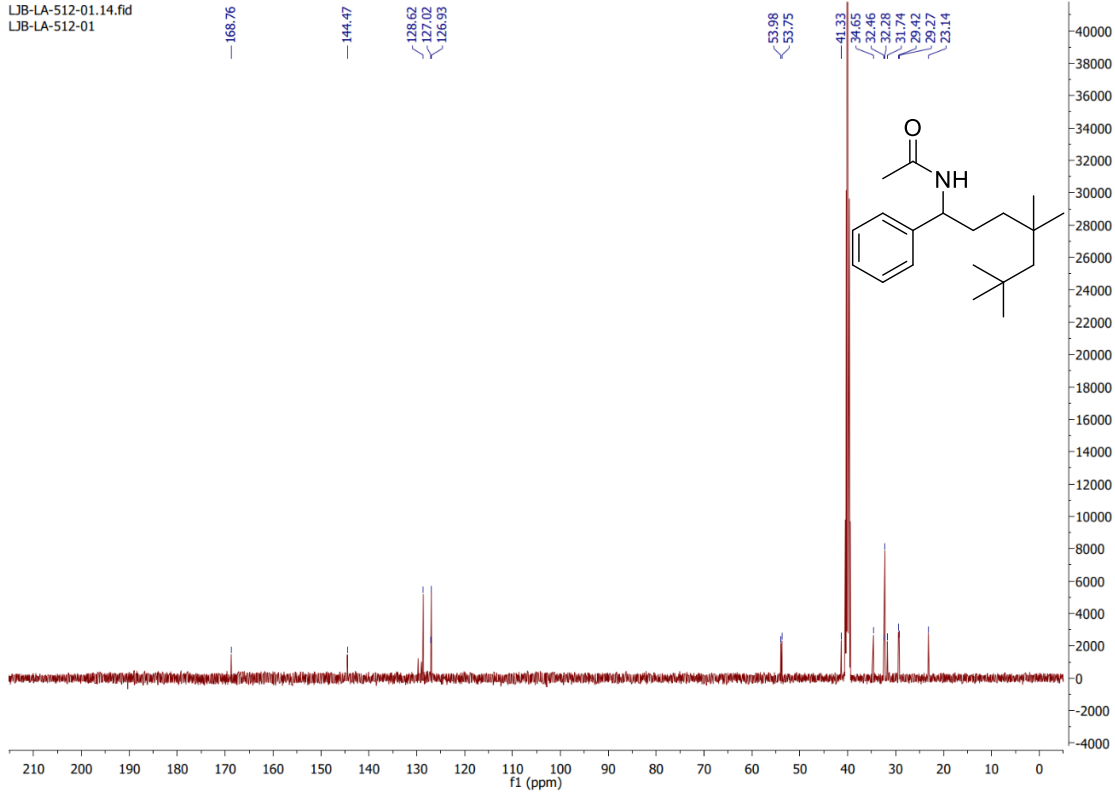
Suggestion:

C16H25N1O1 MW: 247

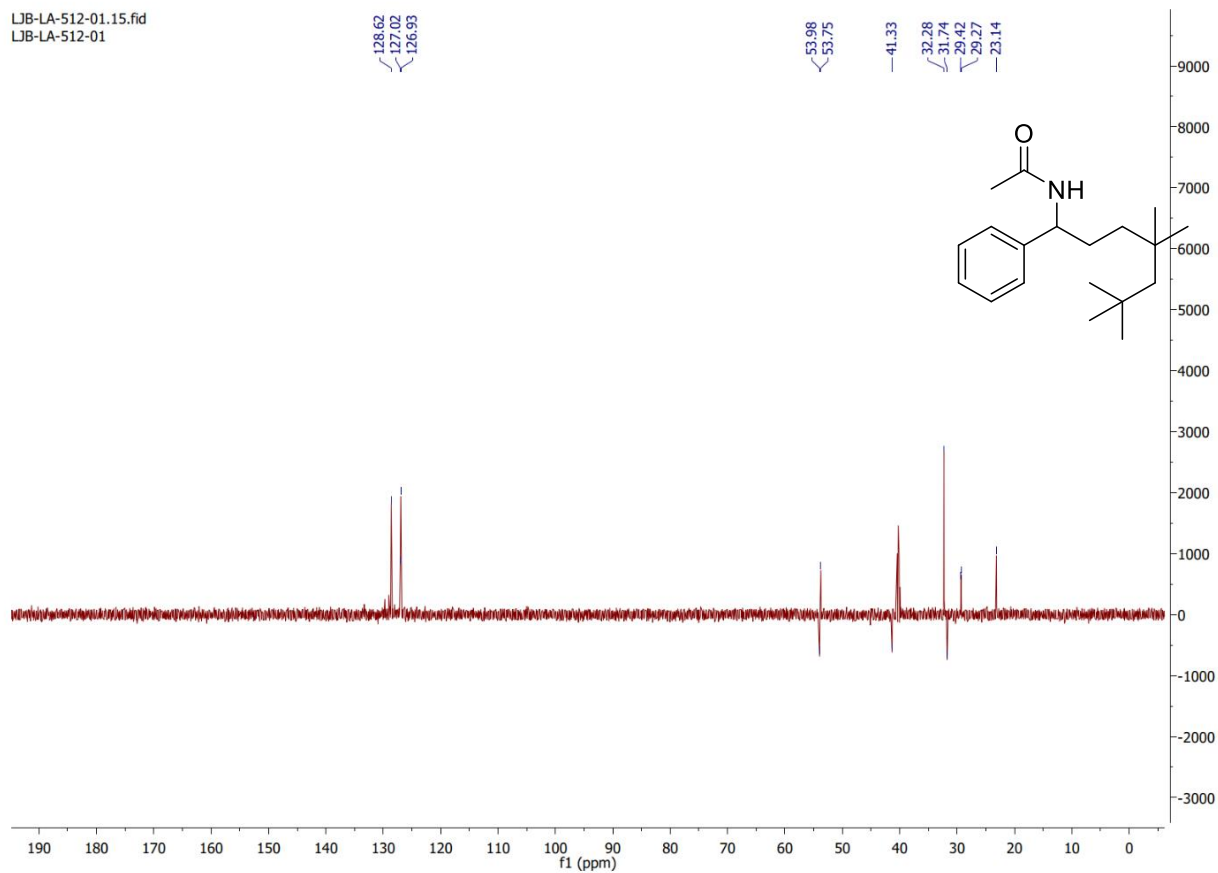
LJB-LA-512-01.12.fid
LJB-LA-512-01

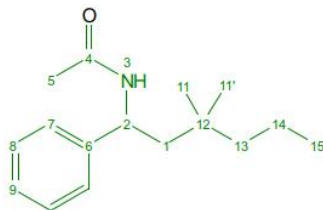


LJB-LA-512-01.14.fid
LJB-LA-512-01



LJB-LA-512-01.15.fid
LJB-LA-512-01





P-ID: CF00204
Measured on: 16/05/2020
CHIFFRE: LJB-LA-521-01
ELNA#: 3972
Client: Sensheng Liu
Group: Klußmann
Spectroscopist: Fares
Analysed on: 18/06/2020
Analysed by: Fares
Amount: 2.0 mg
Solvent: DMSO
Reference: solvent
Temperature: 298 K
Spectrometer: av600a
Probe: cryTCI
Experiments: 1H-zg30, 13C-zgdc30, [13C, 1H]-hsqcedetgpsisp2.4, [13C, 1H]-hmbcetgpl3nd, [1H, 1H]-cosygpmfphpp, [1H, 1H]-noesygpqh, [15N, 1H]-hmbcf3gpndqf, 15N-

Notes:

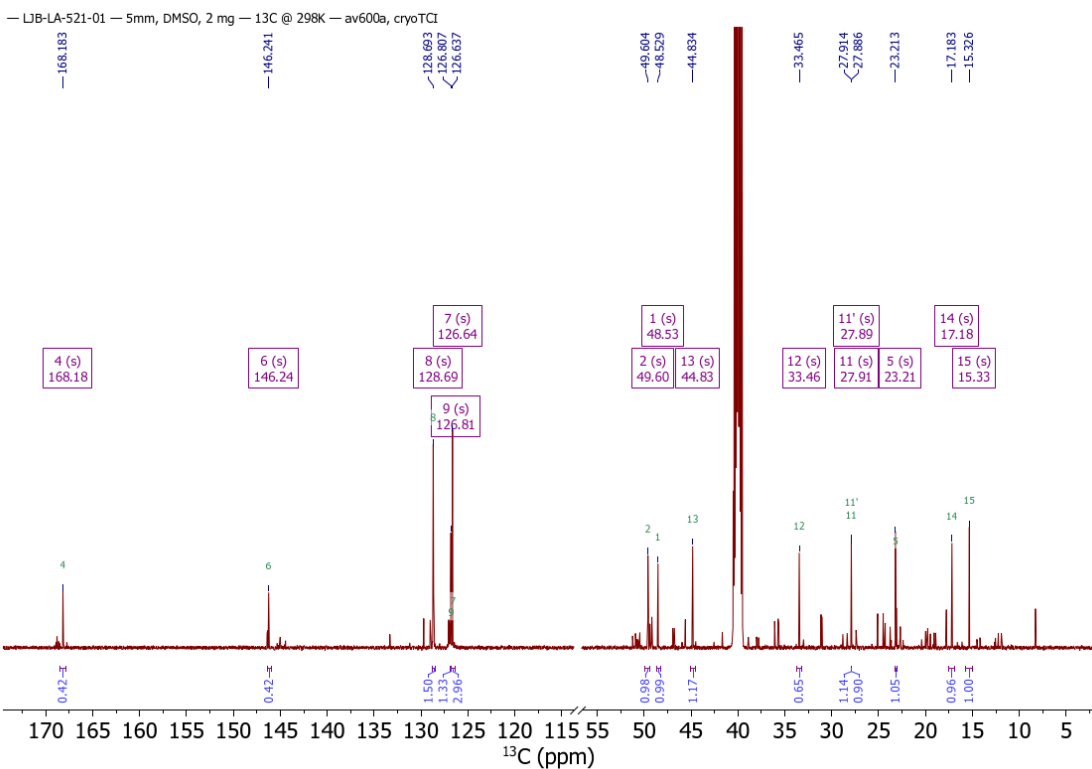
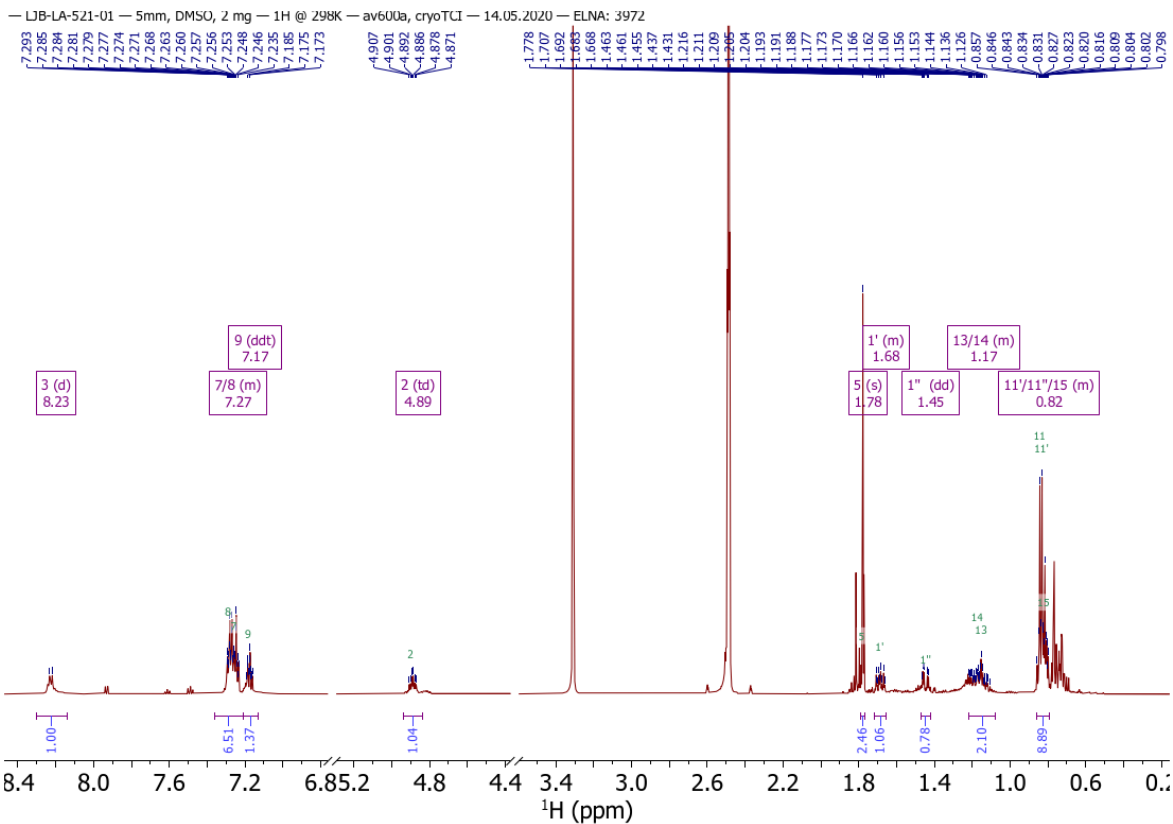
-this sample contains many products but only the major components was assigned.
 -The suggested structure is characteristic of the quaternary carbon C12 and the almost equivalent methyl groups C11 and C11'.
 -Due to overlap and presence of many side-products, the quality of the 1H spectrum did not allow a precise multiplet and integral characterization.
 -The stereochemical assignment of C11 and C11' was not attempted.
 -The NOESY was not evaluated.

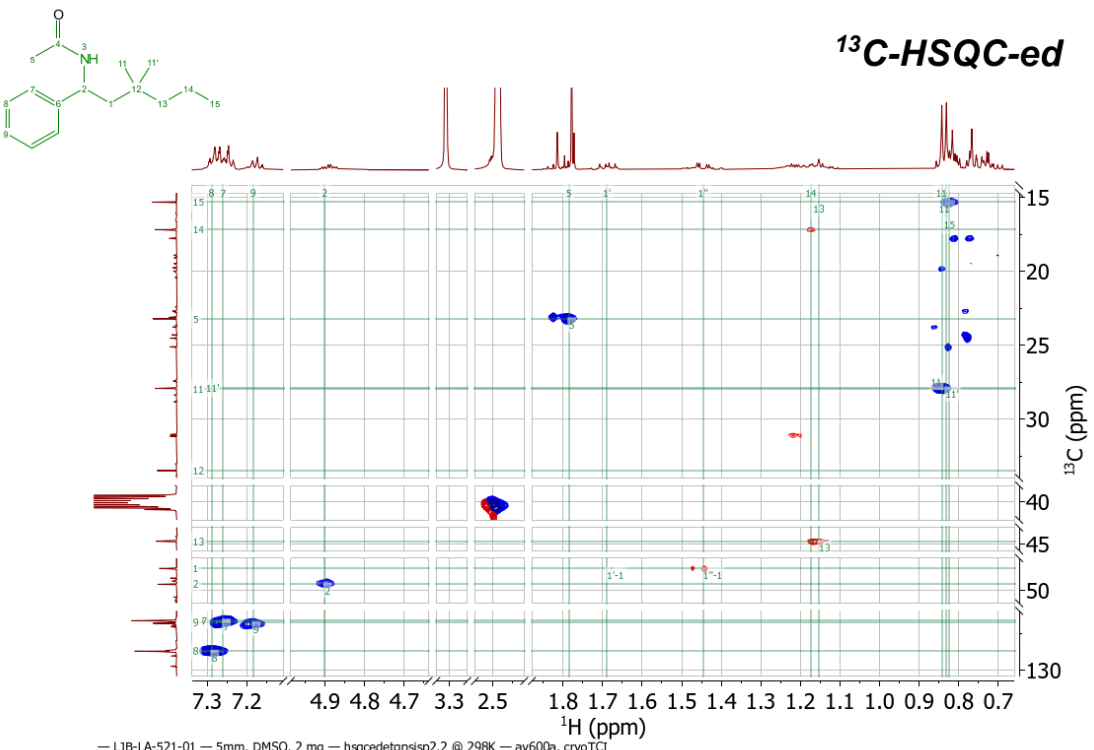
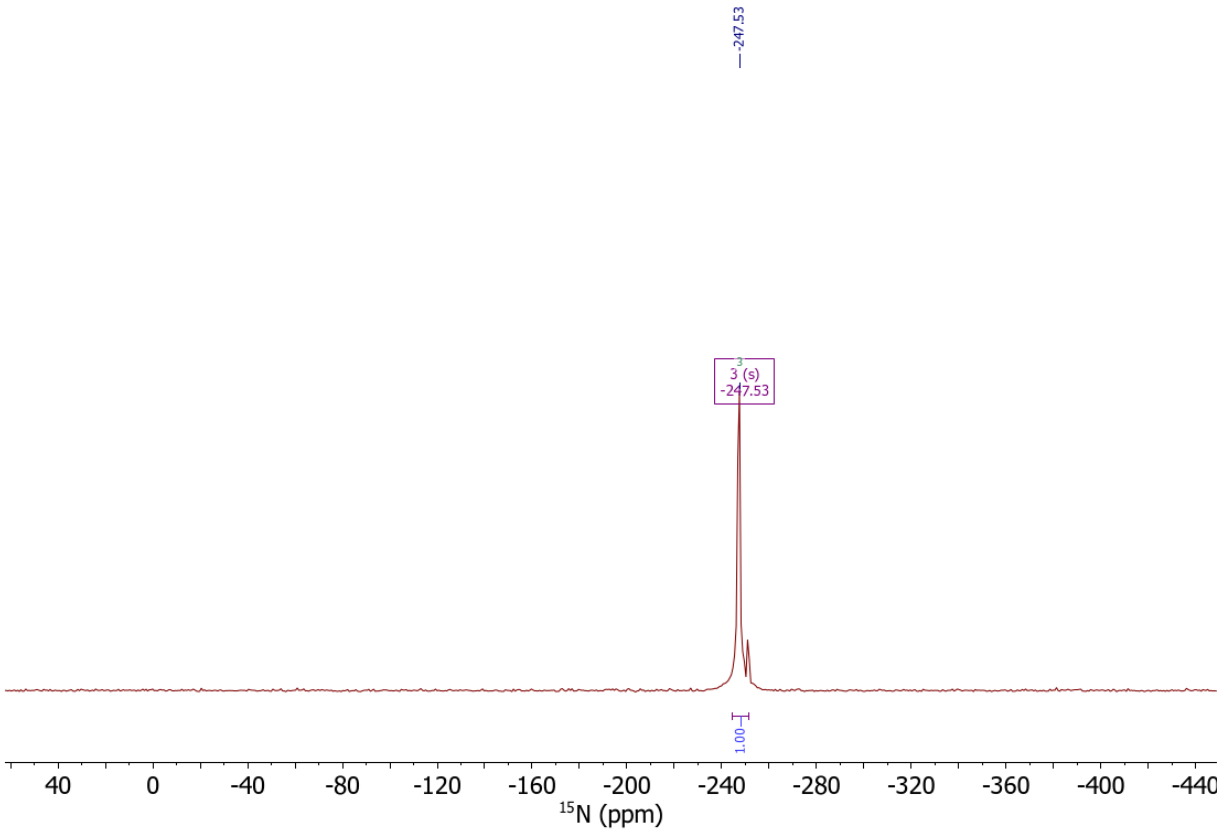
¹H NMR (600 MHz, DMSO) δ 8.23 (d, *J* = 8.6 Hz, 1H, 3), 7.36 – 7.21 (m, 3H, 7/8), 7.17 (ddt, *J* = 7.2, 5.3, 1.4 Hz, 1H, 9), 4.89 (td, *J* = 9.1, 4.1 Hz, 1H, 2), 1.78 (s, 3H, 5), 1.72 – 1.65 (m, 1H, 1'), 1.45 (dd, *J* = 14.3, 3.6 Hz, 1H, 1''), 1.22 – 1.08 (m, 4H, 13/14), 0.86 – 0.79 (m, 9H, 11/11'/15).

¹³C NMR (151 MHz, DMSO) δ 168.18 (4), 146.24 (6), 128.69 (8), 126.81(9), 126.64(7), 49.60 (2), 48.53 (1), 44.83 (13), 33.46 (12), 27.91), 27.89 (11'), 23.21), 17.18 (14), 15.33 (15).

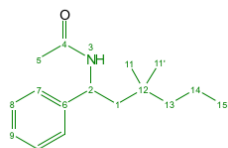
¹⁵N NMR (61 MHz, DMSO) δ -247.53, 3.

| Atom | δ (ppm) | Predicted Shift | COSY | HSQC | HMBC |
|-------|---------|-----------------|---------|---------|--------------------------|
| 1 C | 48.53 | 47.3 | | 1', 1'' | 2, 11, 11' |
| H | 1.69 | 1.78, 1.81 | 1'', 2 | 1 | 2, 3, 6, 11, 11', 12, 13 |
| H' | 1.45 | 1.78, 1.81 | 1', 2 | 1 | 3 |
| 2 C | 49.6 | 53.46 | | 2 | 1' |
| H | 4.9 | 4.94 | 1', 1'' | 2 | 1, 6, 7, 12 |
| 3 N | -247.53 | | | | 1', 1'', 5 |
| H | | 6.61 | | | |
| 4 C | 168.18 | 170.68 | | | 5 |
| 5 C | 23.21 | 23.31 | | 5 | |
| H3 | 1.79 | 1.88 | | 5 | 3, 4 |
| 6 C | 146.24 | 143.21 | | | 1', 2, 8 |
| 7 C | 126.58 | 126.31 | | 7 | 2, 7, 9 |
| H | 7.26 | 7.32 | | 7 | 7, 9 |
| 8 C | 128.69 | 128.32 | | 8 | 8 |
| H | 7.29 | 7.32 | | 8 | 6, 8 |
| 9 C | 126.77 | 127.47 | | 9 | 7 |
| H | 7.18 | 7.27 | | 9 | 7 |
| 11 C | 27.91 | 29.14 | | 11 | 1' |
| H3 | 0.84 | 0.99 | | 11 | 1, 11', 12, 13 |
| 11' C | 27.89 | 29.14 | | 11' | 1', 11, 11' |
| H3 | 0.83 | 0.86 | | 11' | 1, 11', 12, 13 |
| 12 C | 33.46 | 34.85 | | | 1', 2, 11, 11' |
| 13 C | 44.83 | 44.44 | | 13 | 1', 11, 11' |
| H2 | 1.15 | 1.27, 1.28 | 14 | 13 | |
| 14 C | 17.18 | 18.13 | | | |
| H2 | 1.17 | 1.27, 1.28 | 13 | | |
| 15 C | 15.33 | 15.08 | | | |
| H3 | 0.82 | 0.88 | | | |

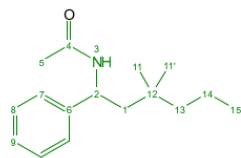
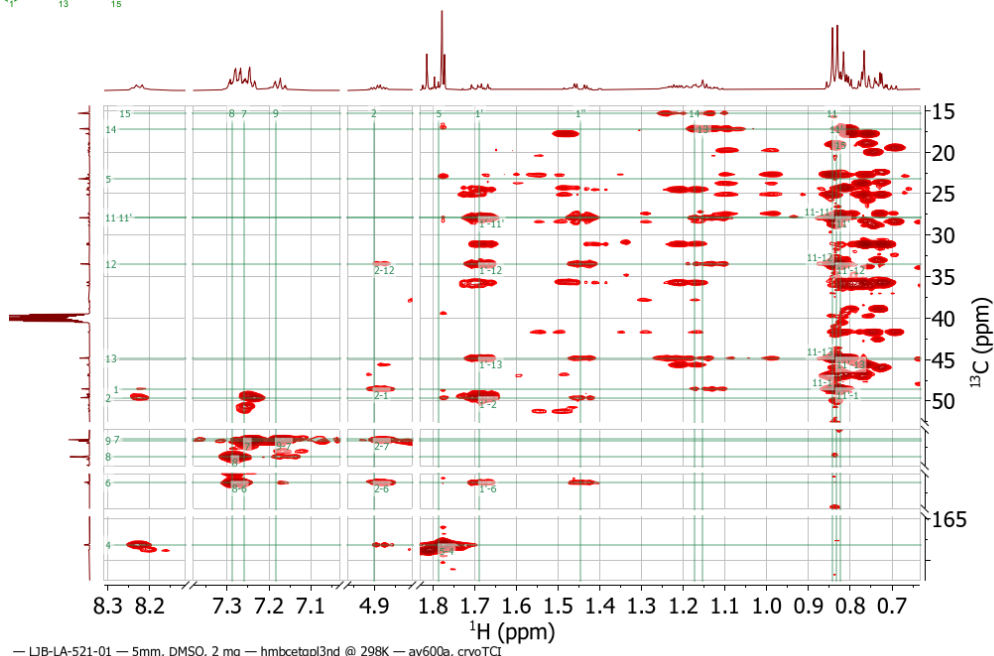




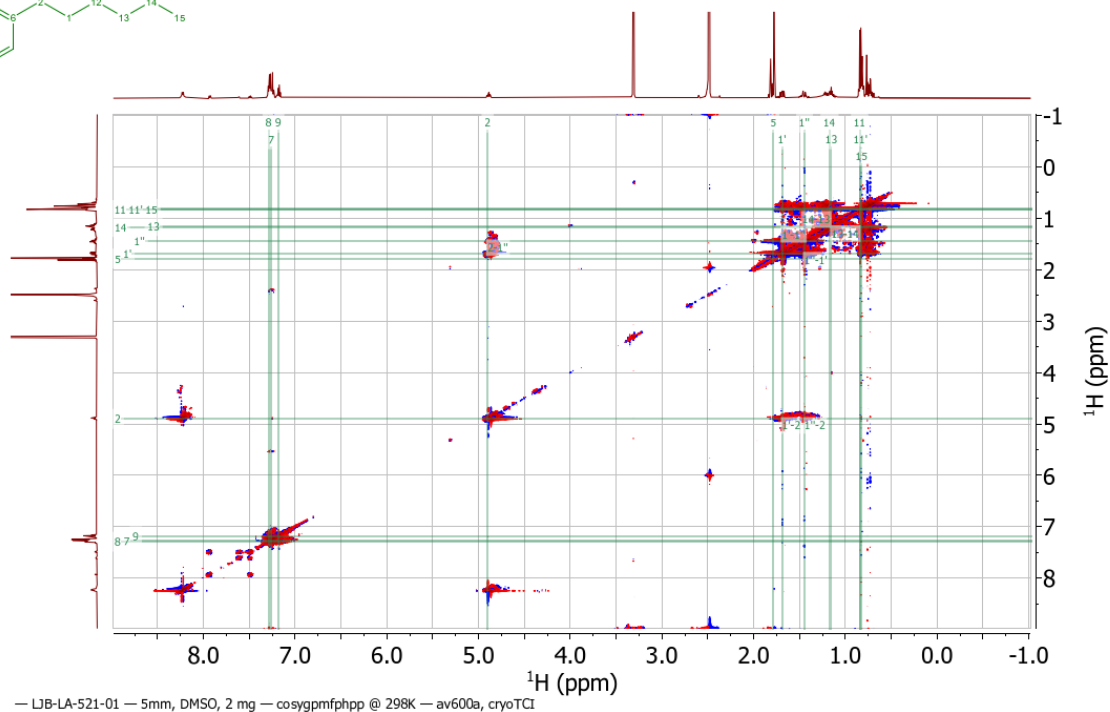
— LJB-LA-521-01 — 5mm, DMSO, 2 mg — hsqcedetgppisp2.2 @ 298K — av600a, cryoTCl

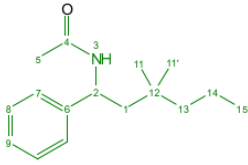


¹³C-HMBC

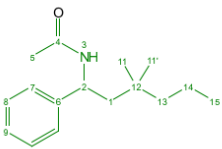
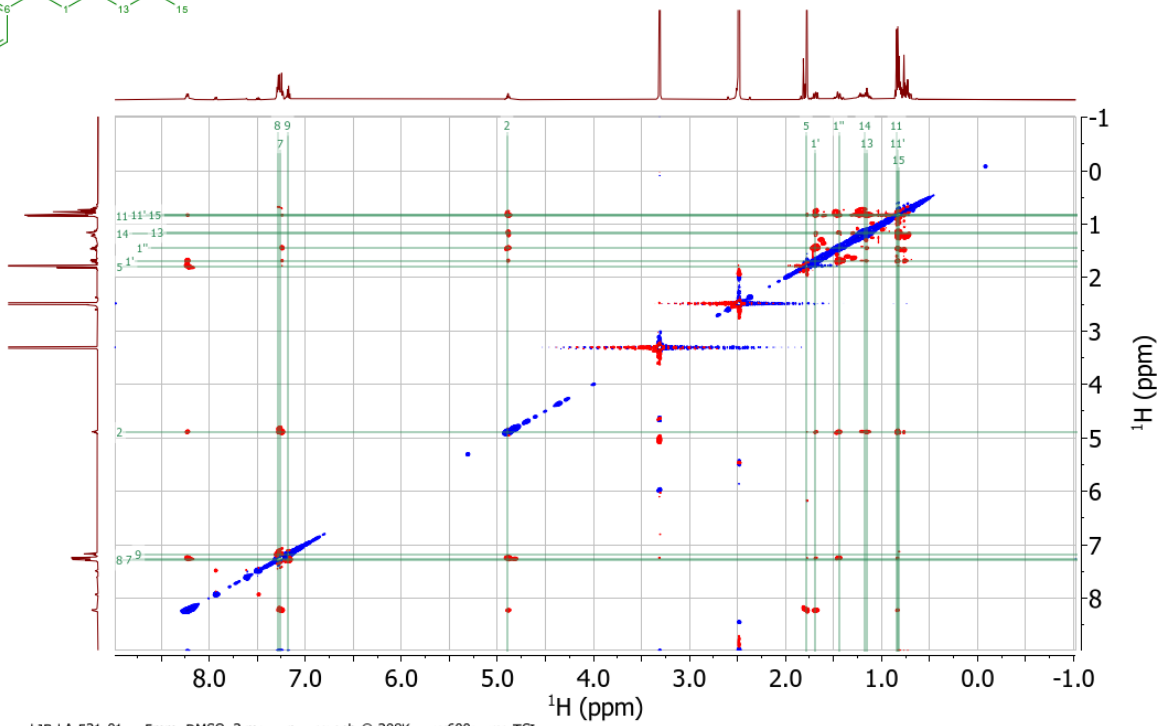


COSY

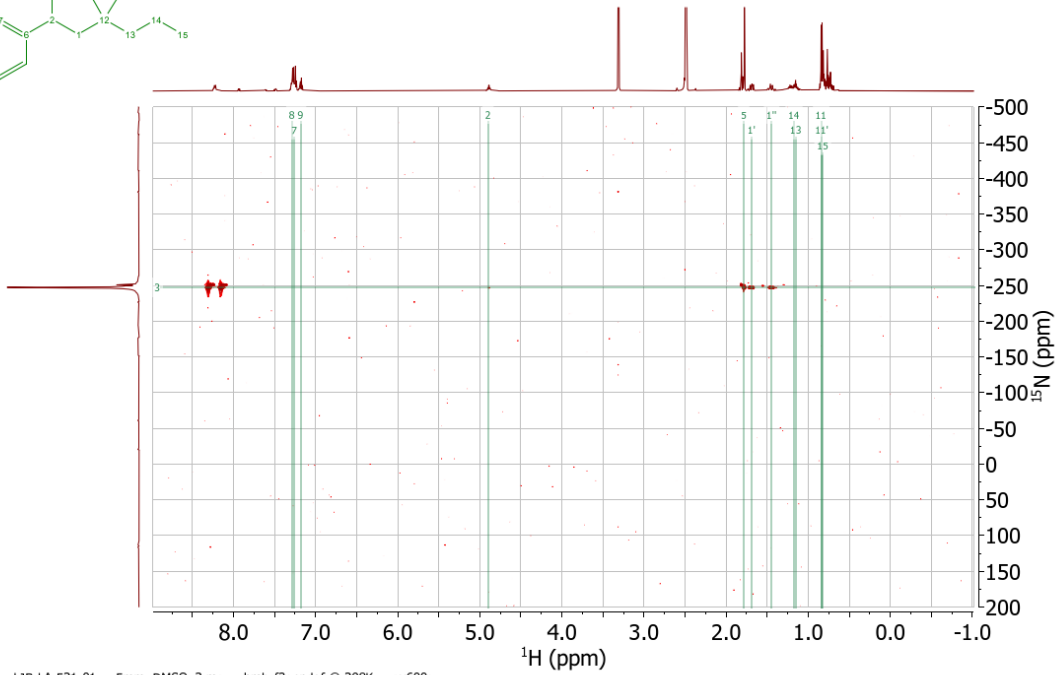


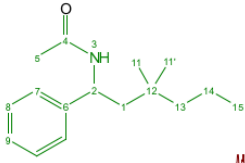


NOESY

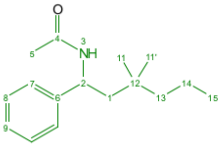
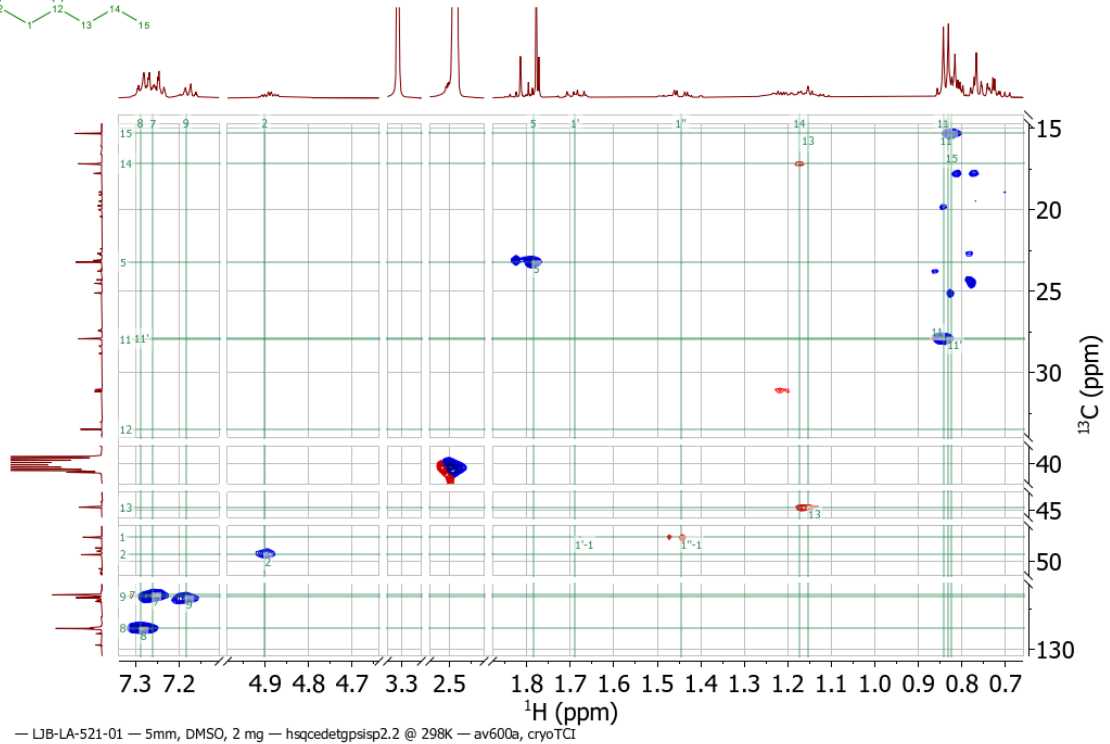


¹⁵N-HMBC

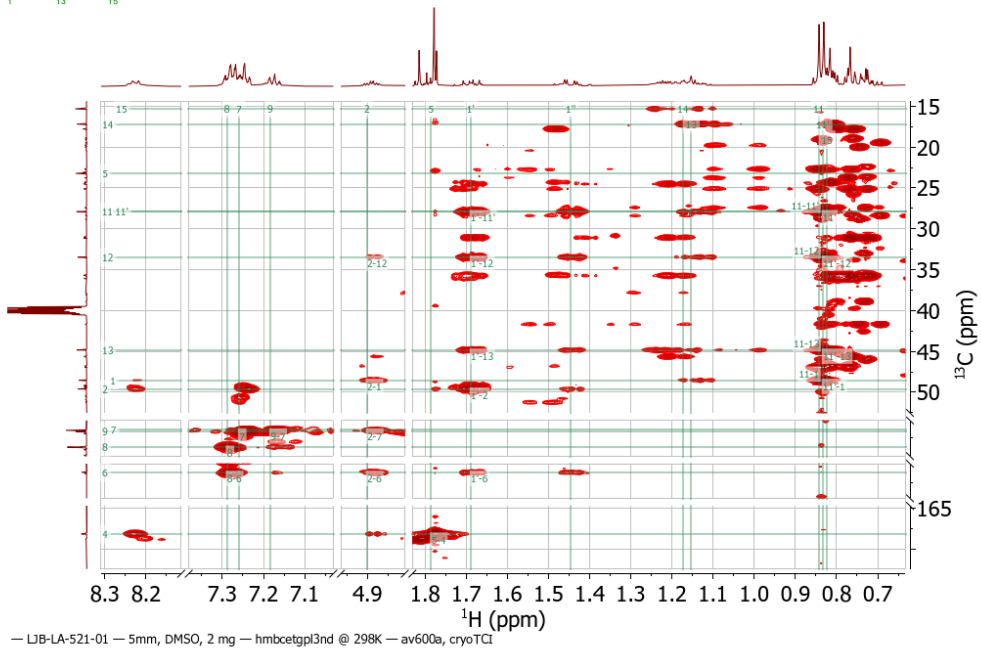


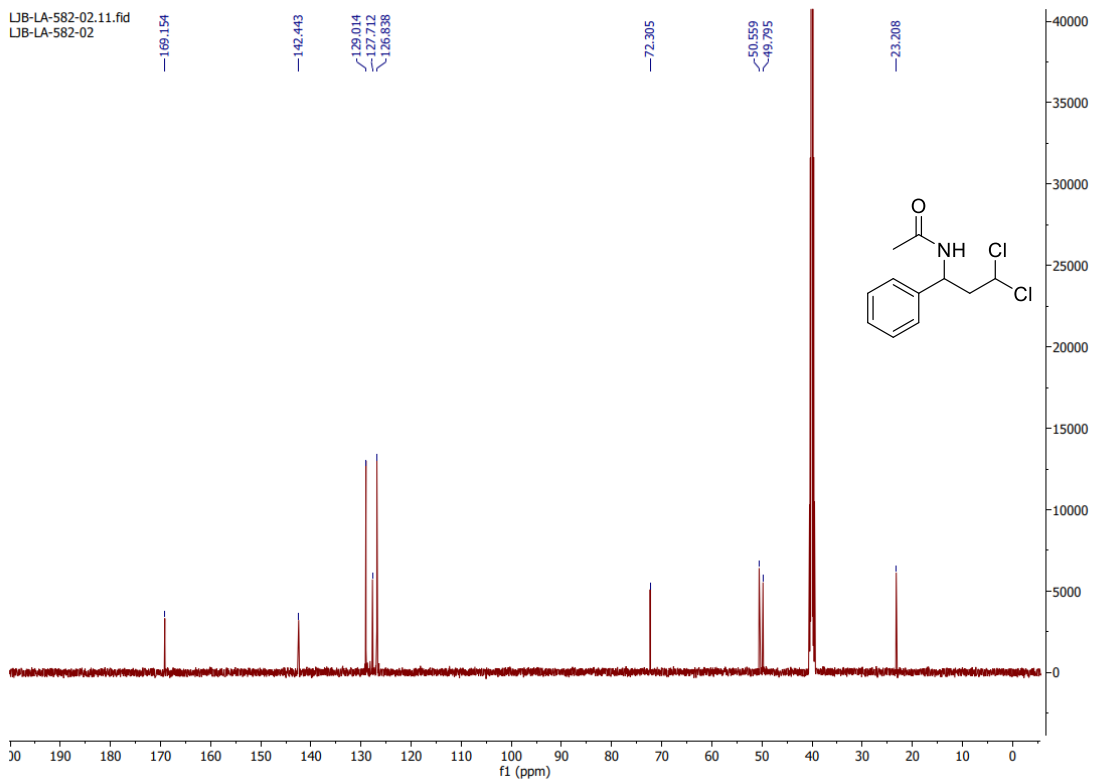
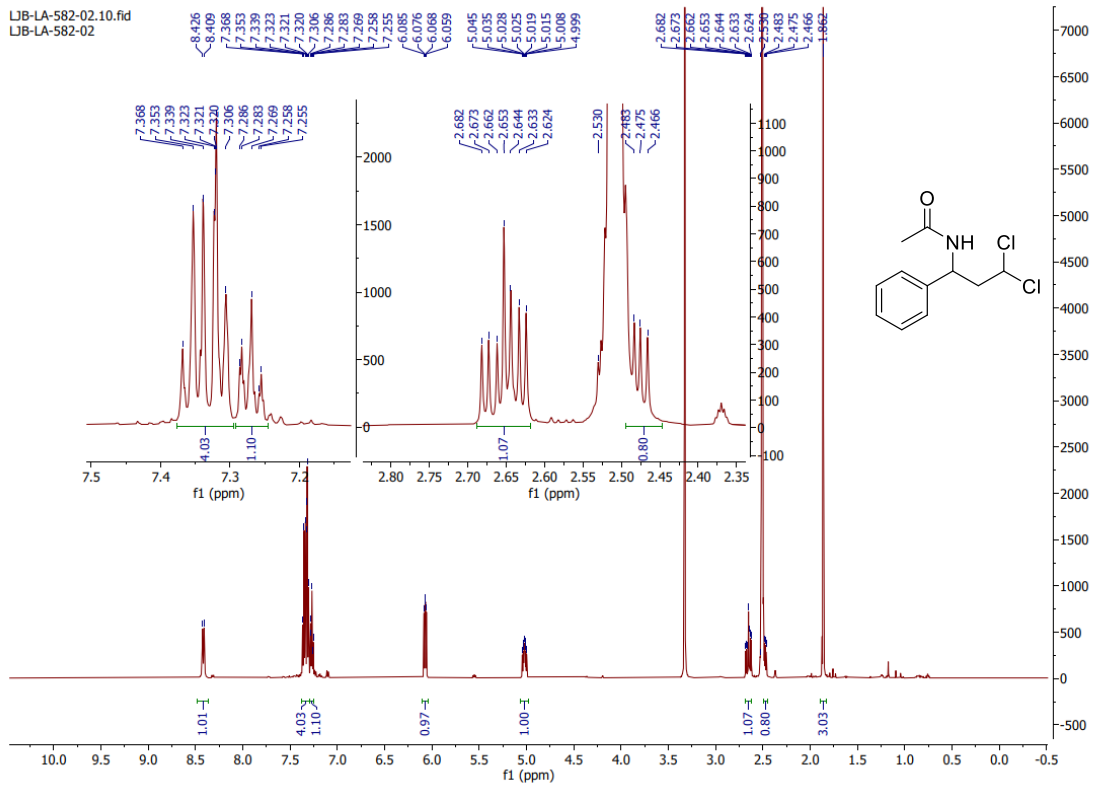


¹³C-HSQC-ed

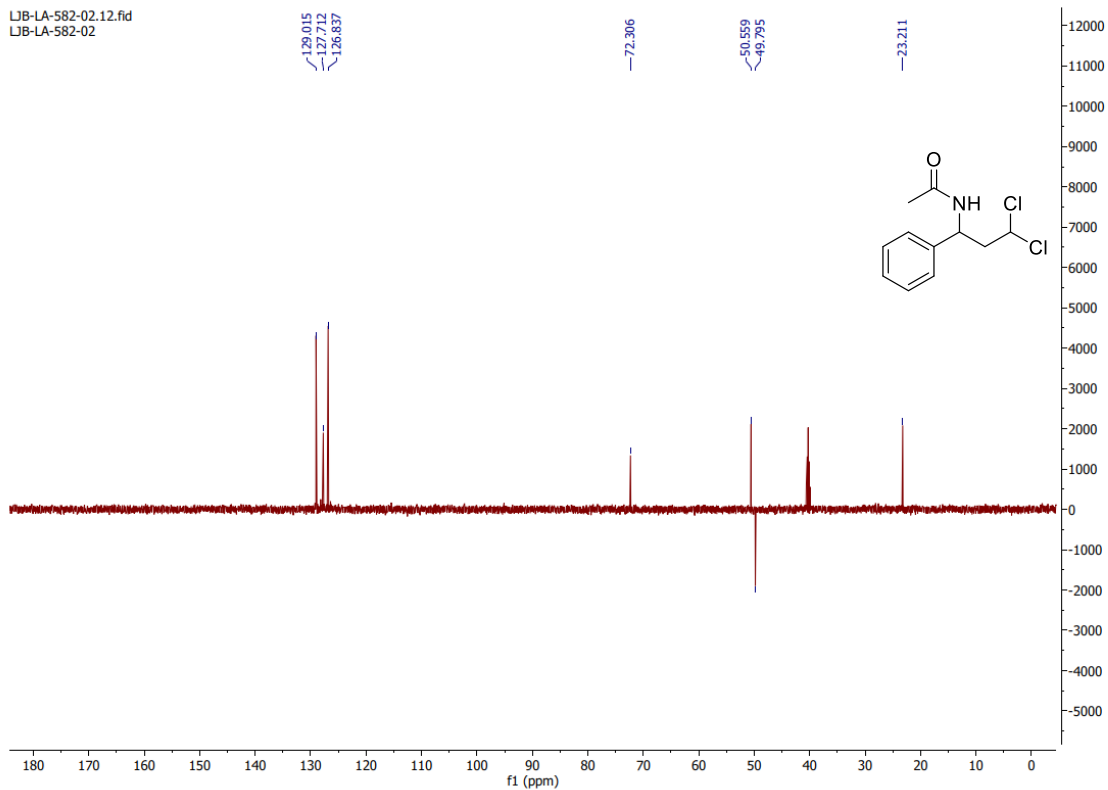


¹³C-HMBC

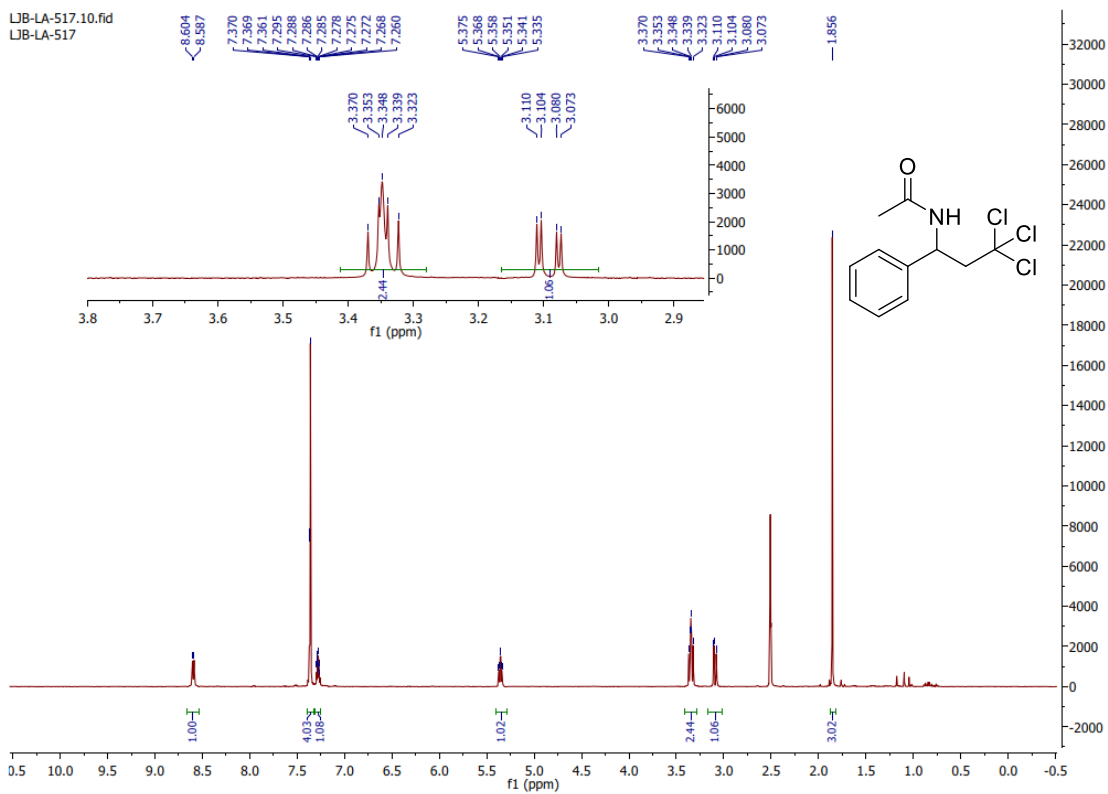


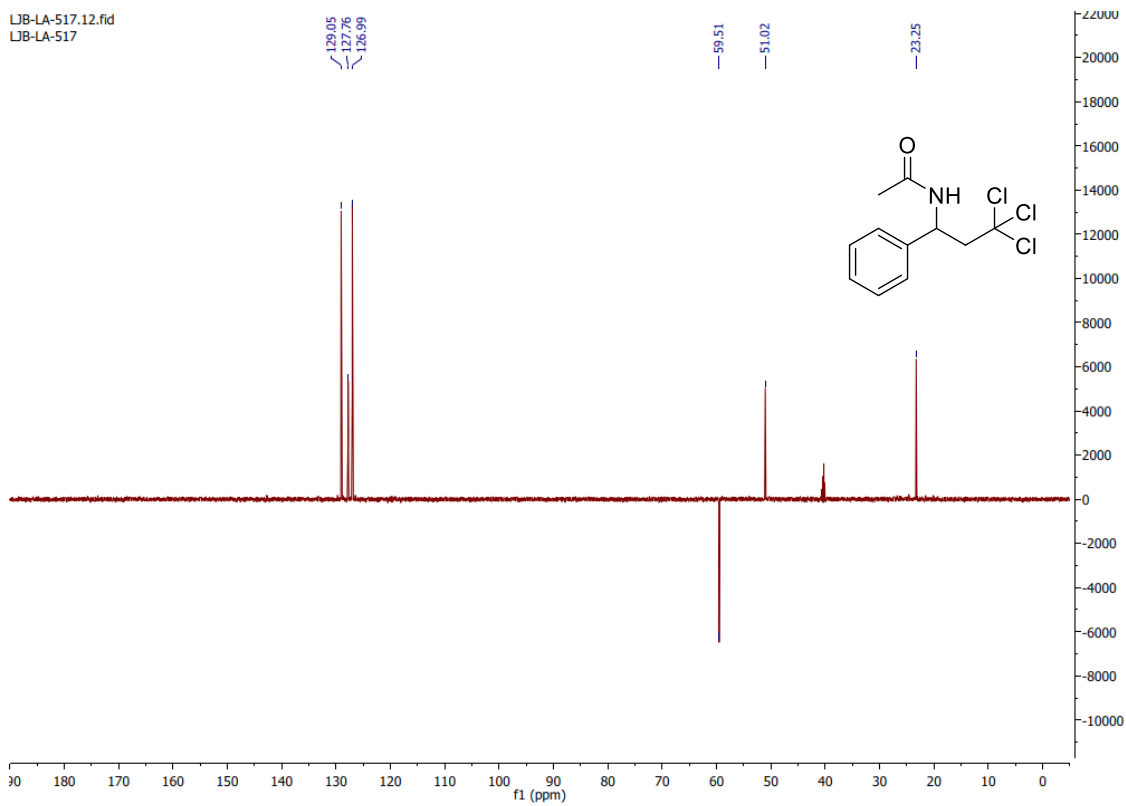
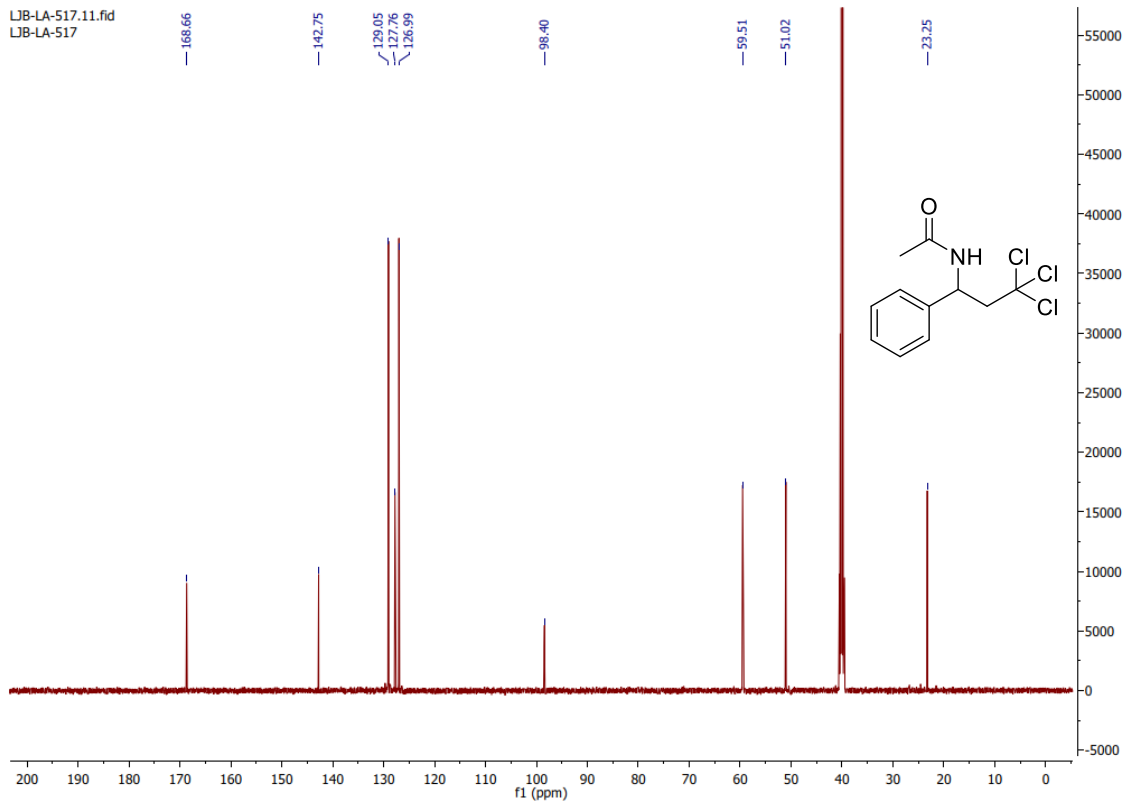


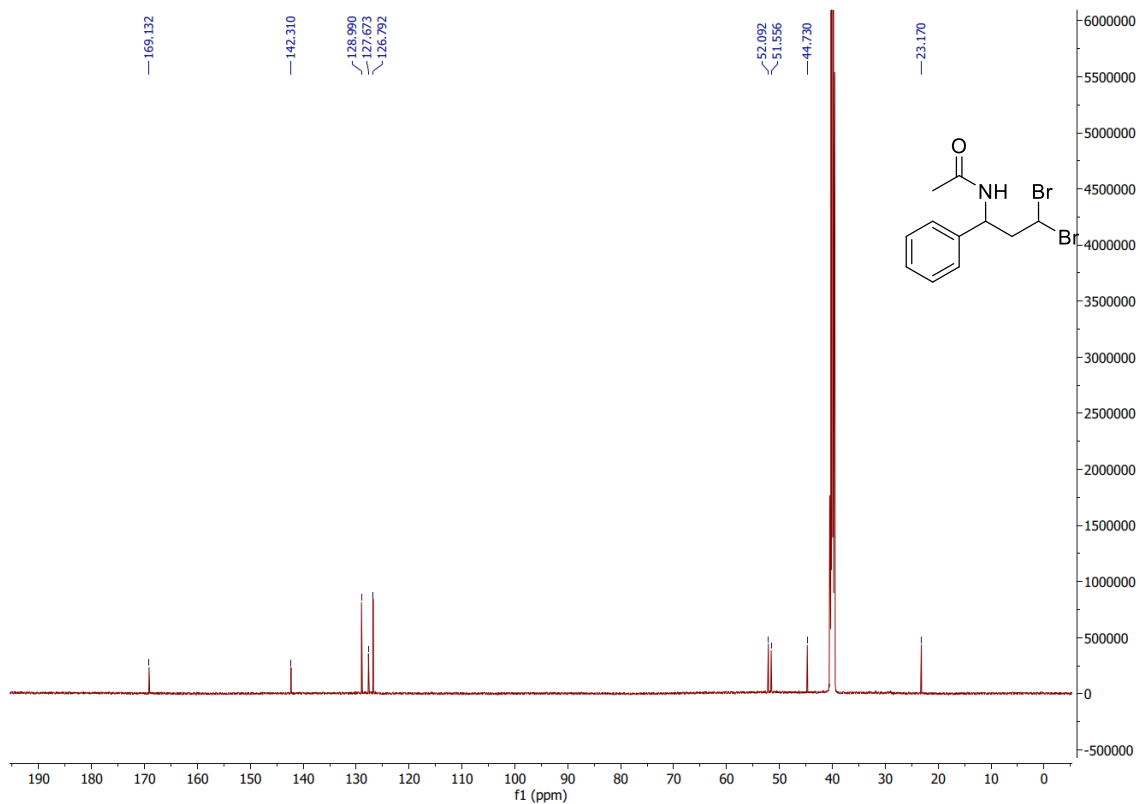
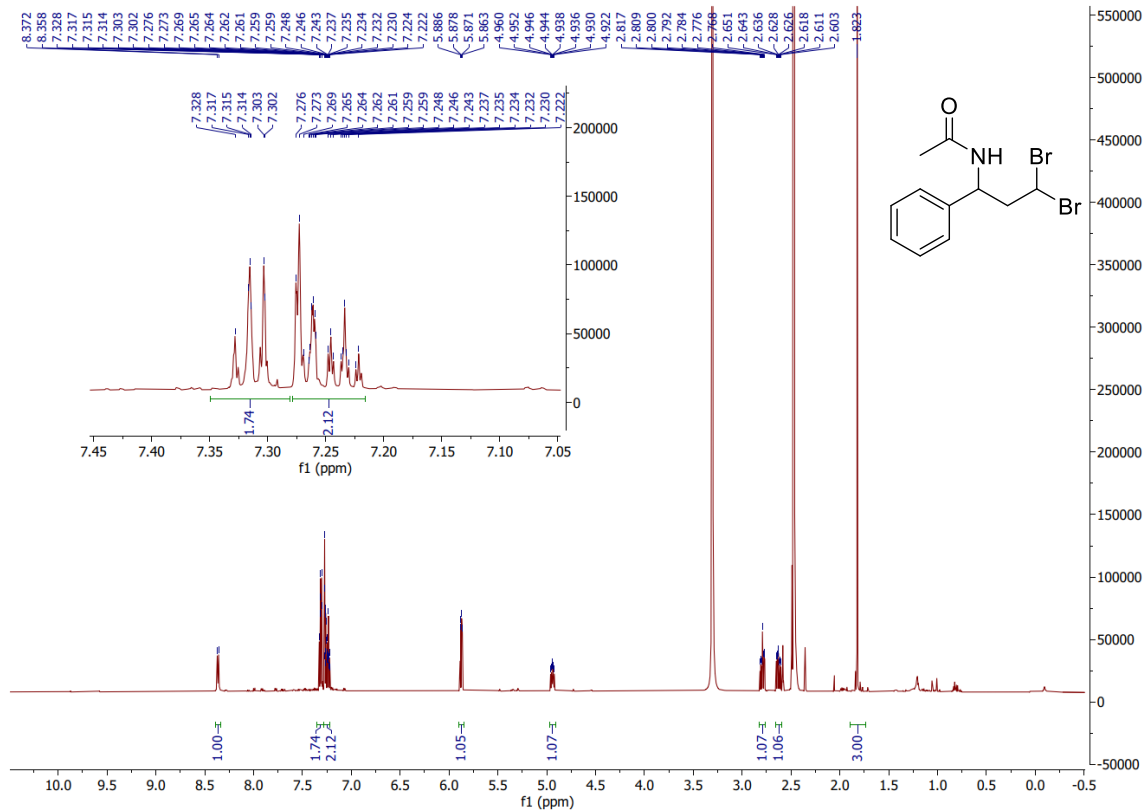
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LJB-LA-582-02

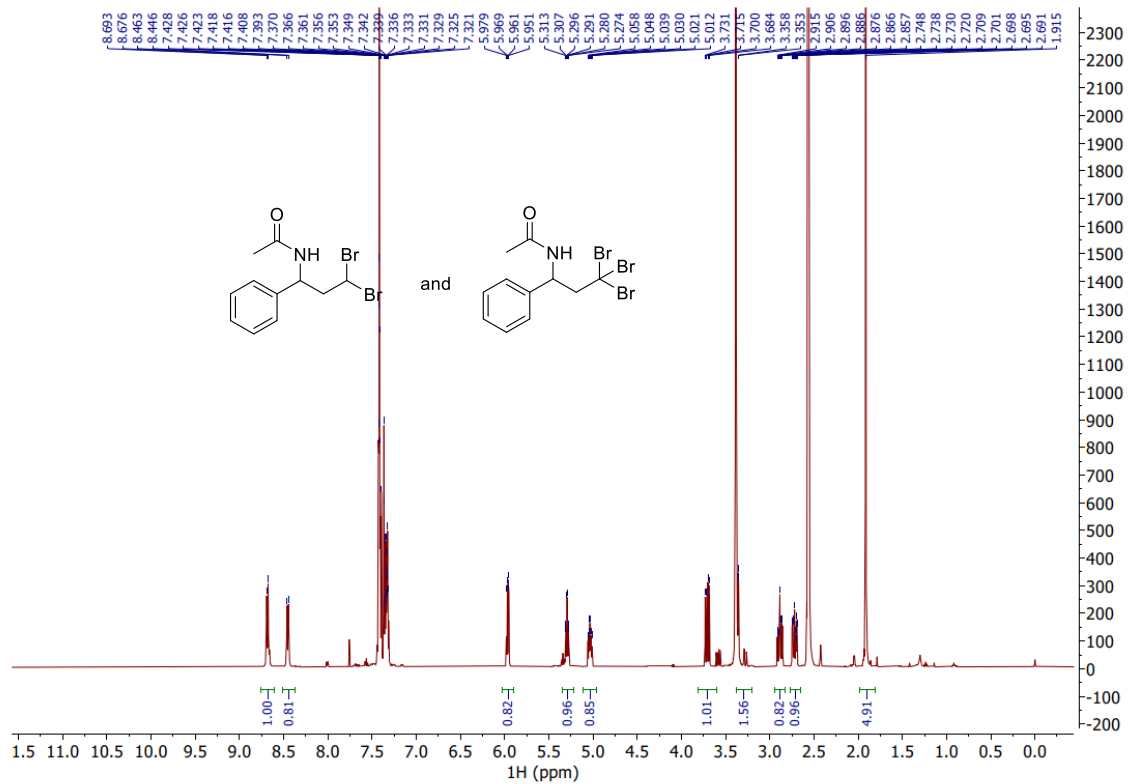
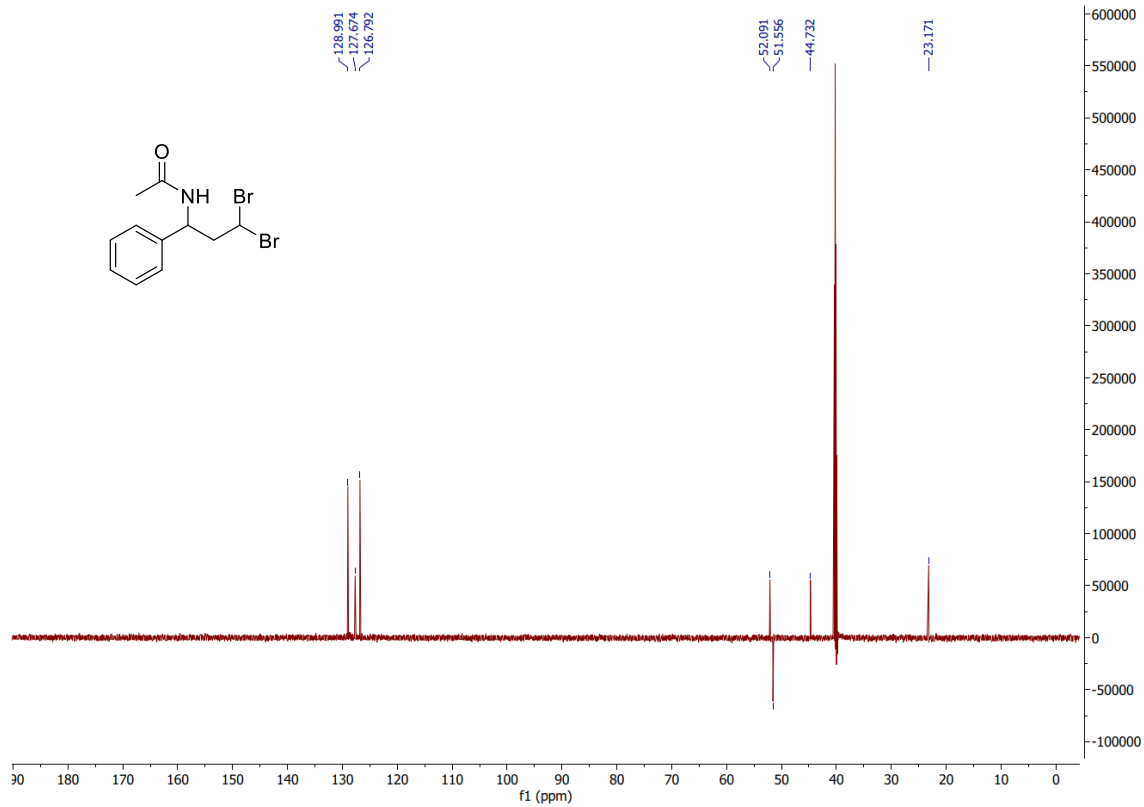


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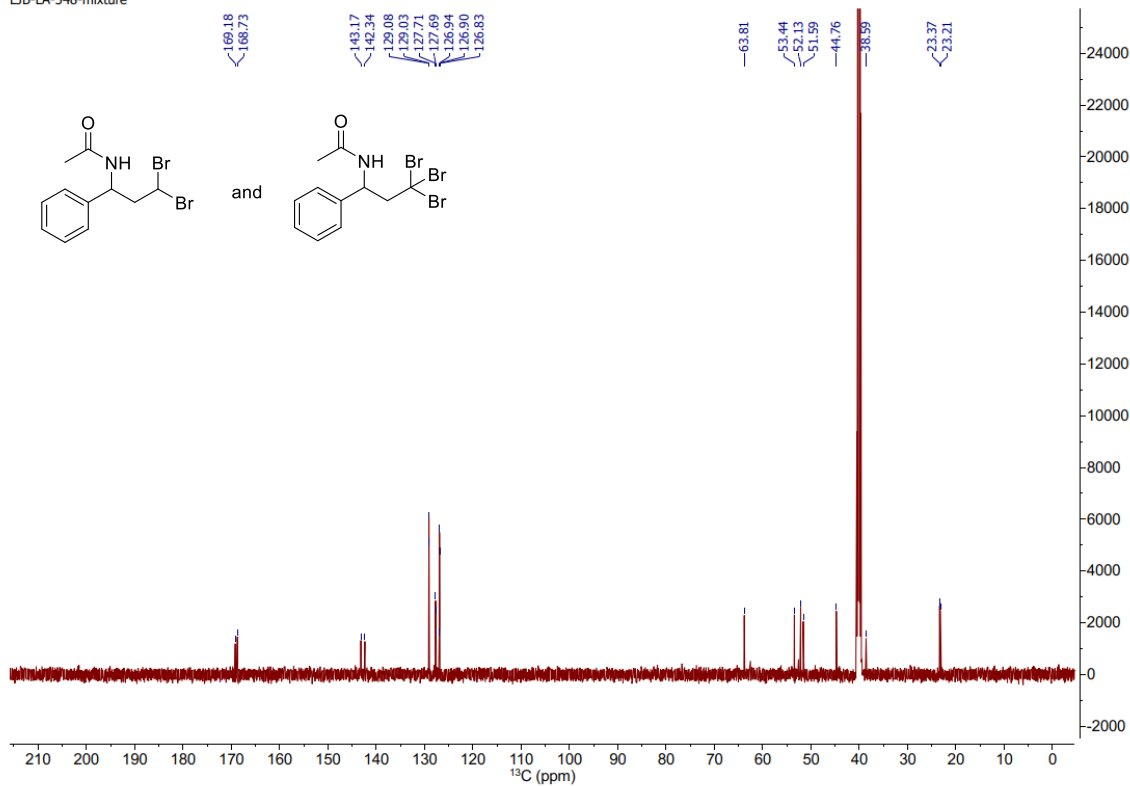




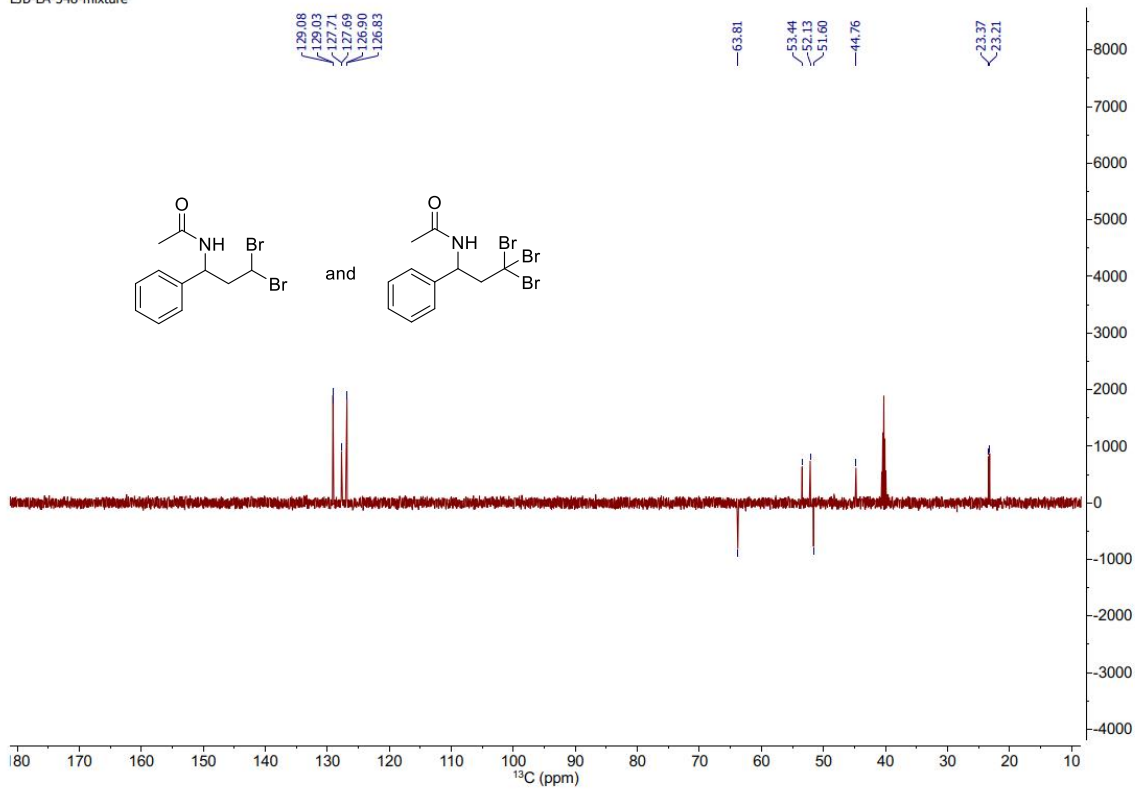


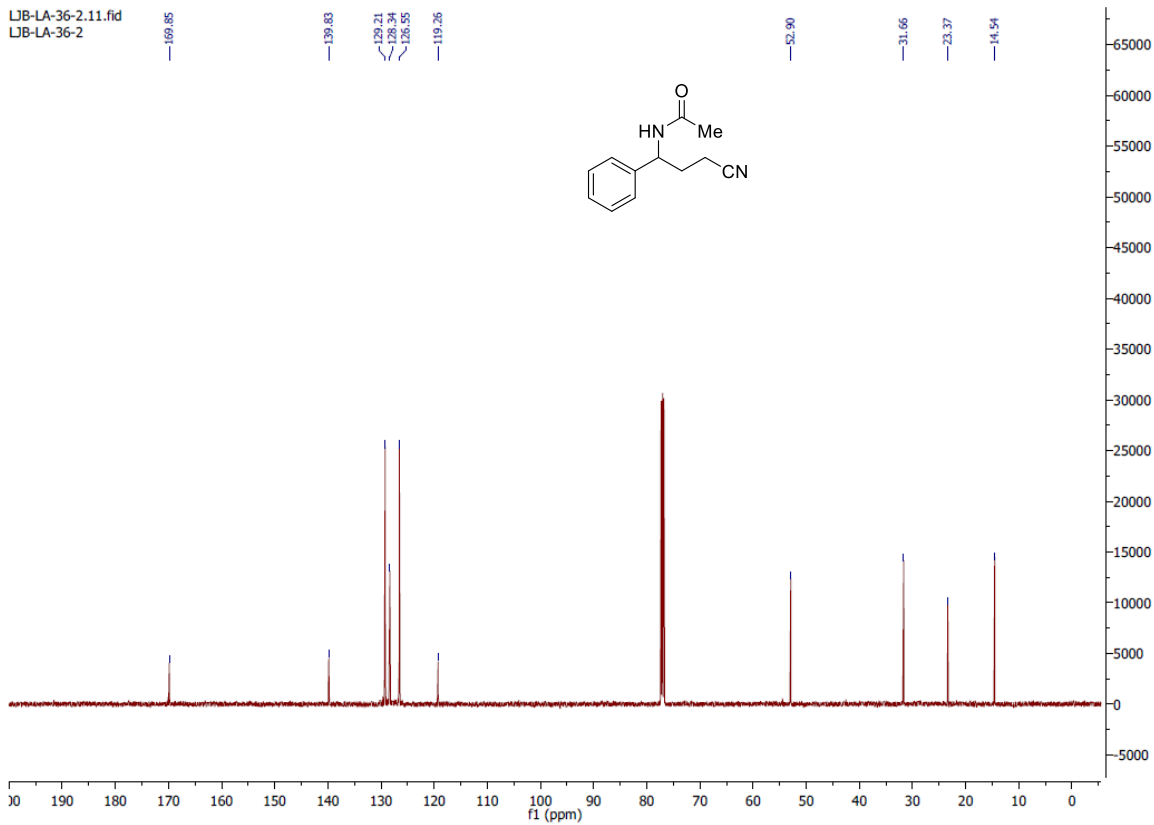
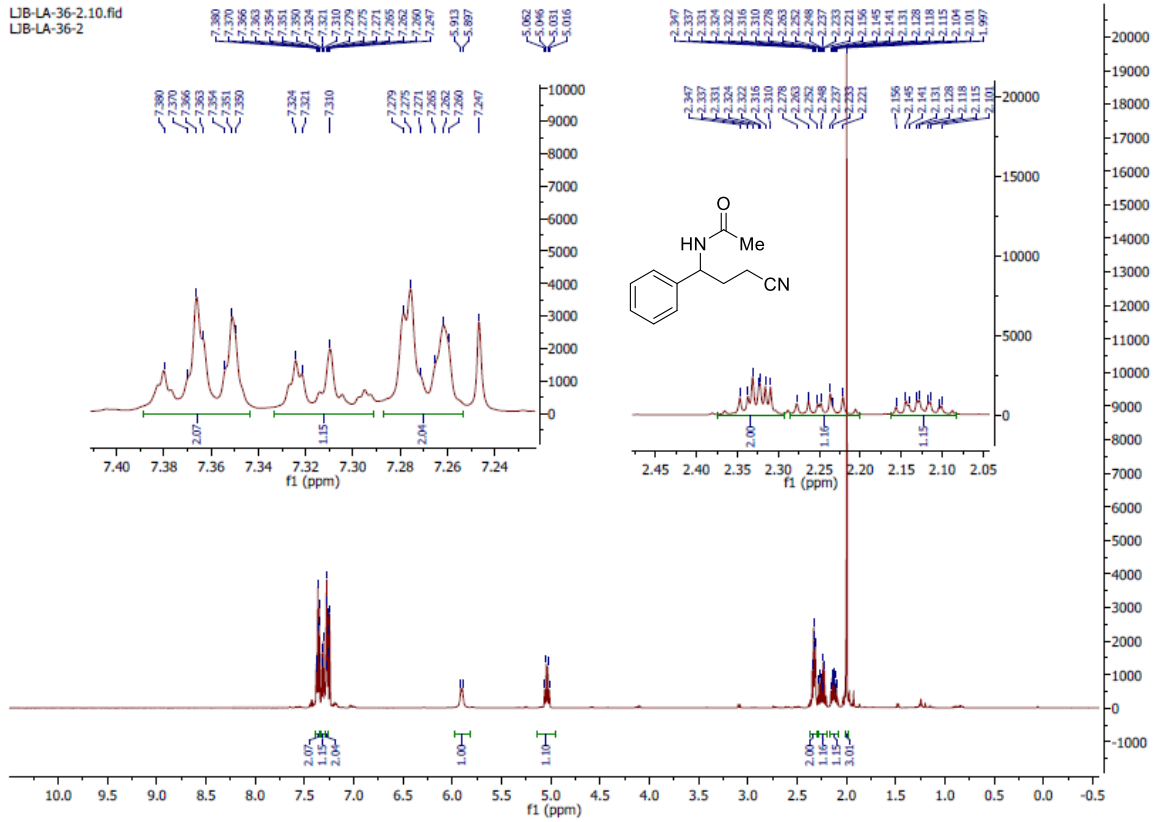


LJB-LA-548-mixture

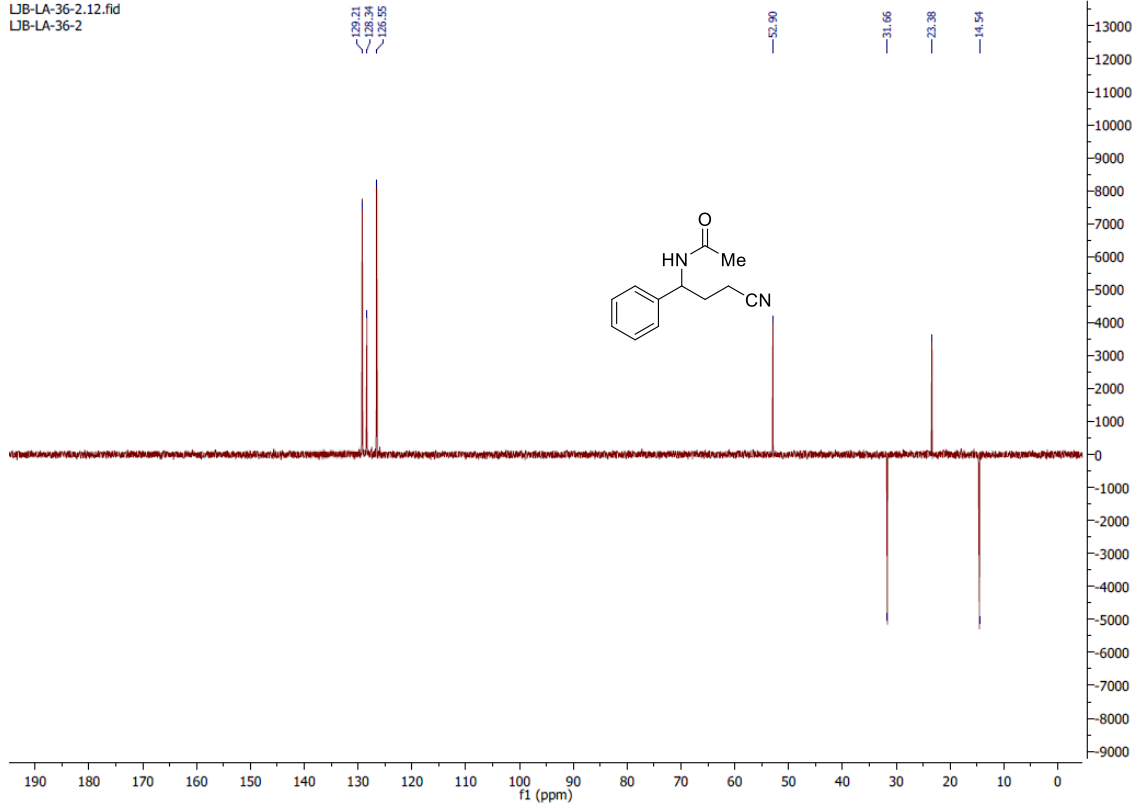


LJB-LA-548-mixture

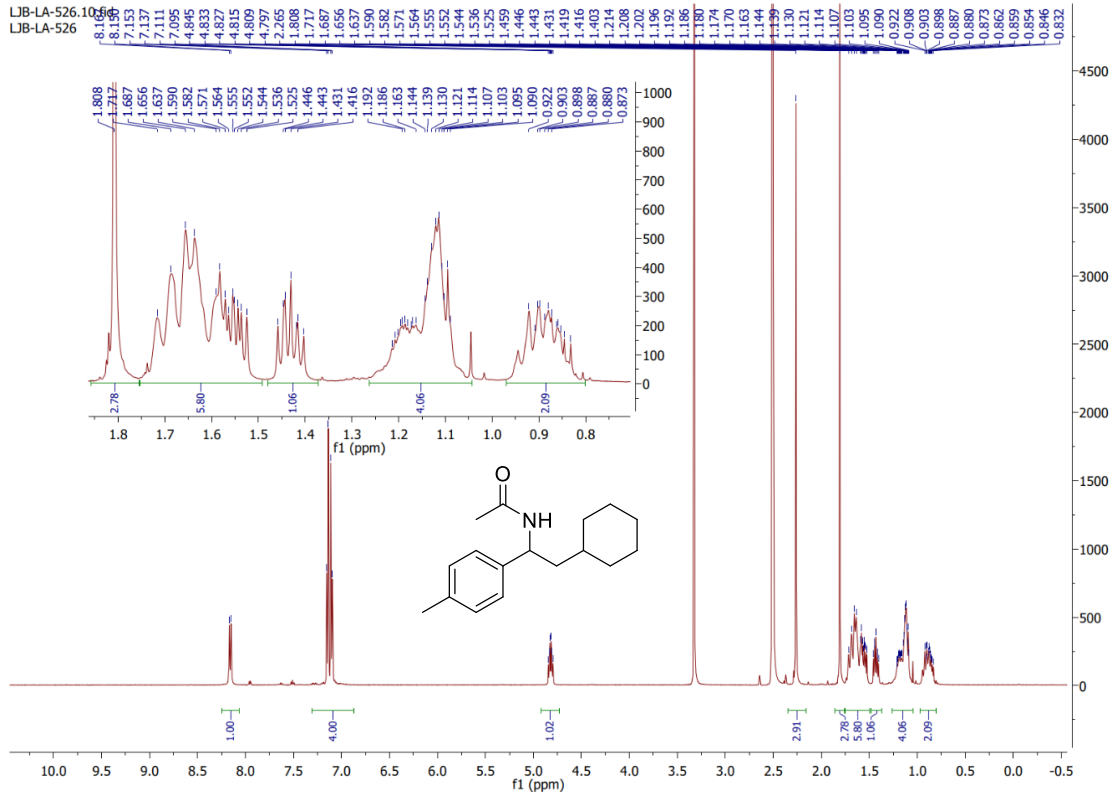


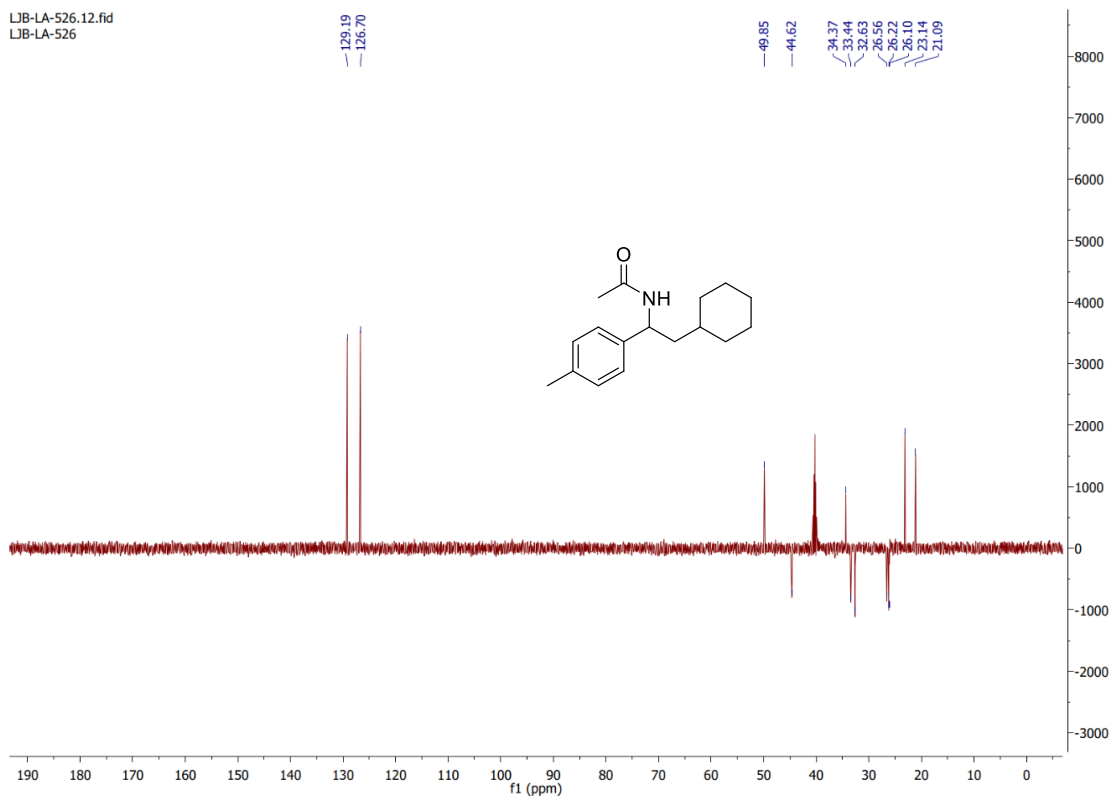
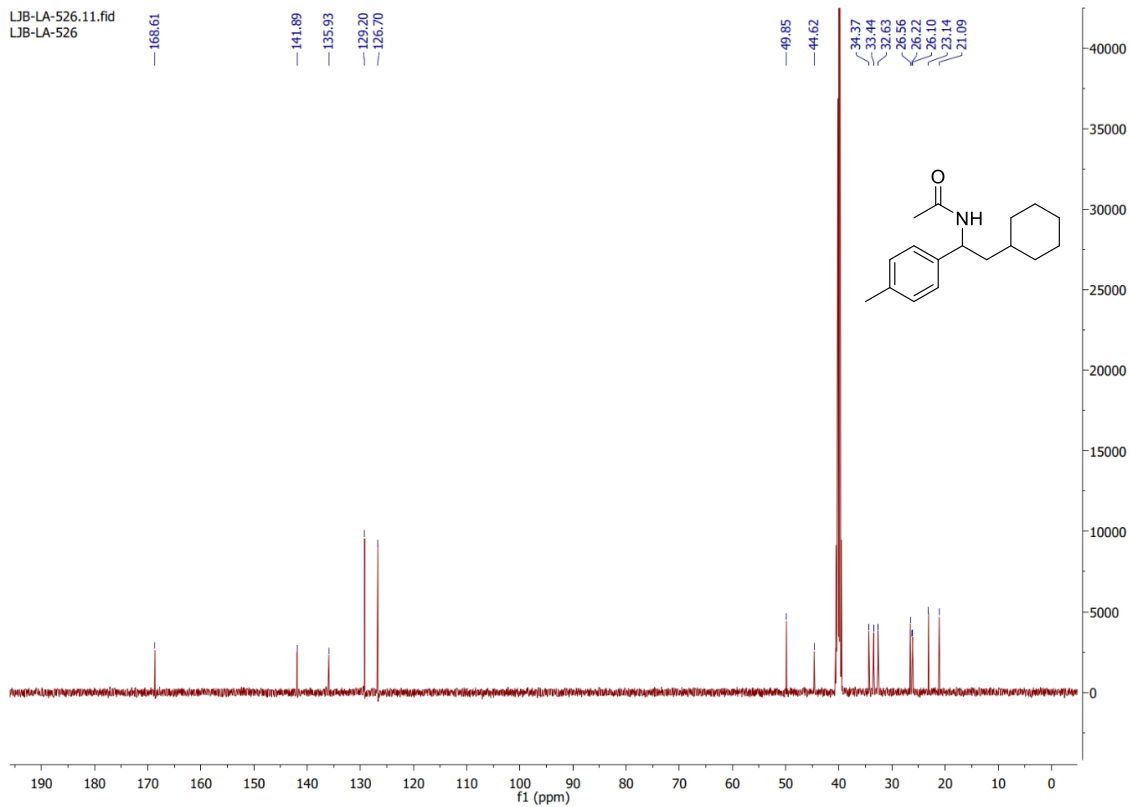


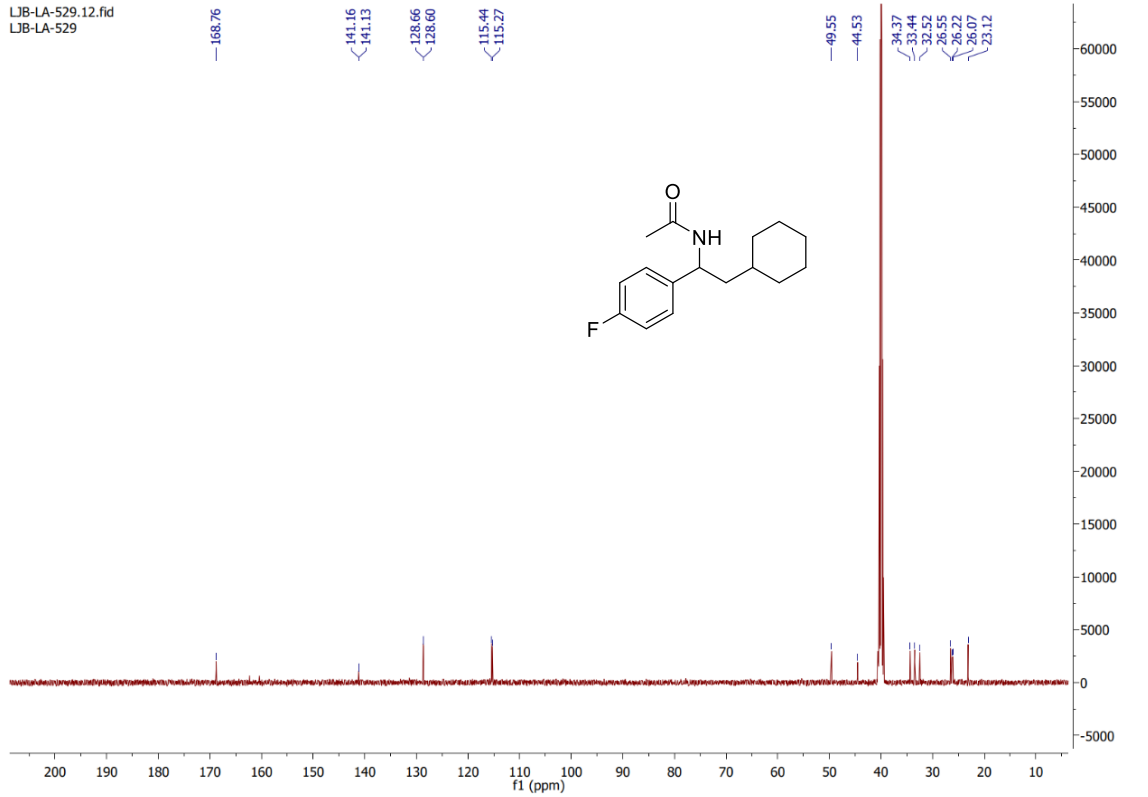
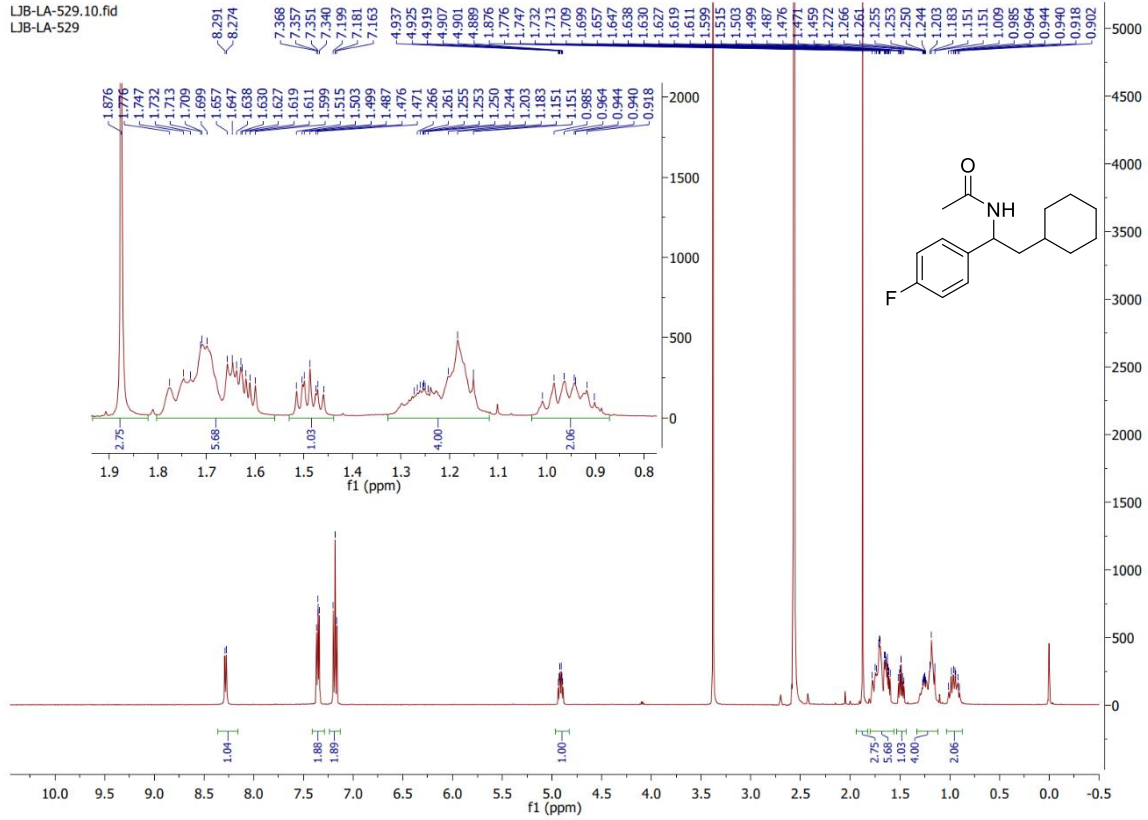
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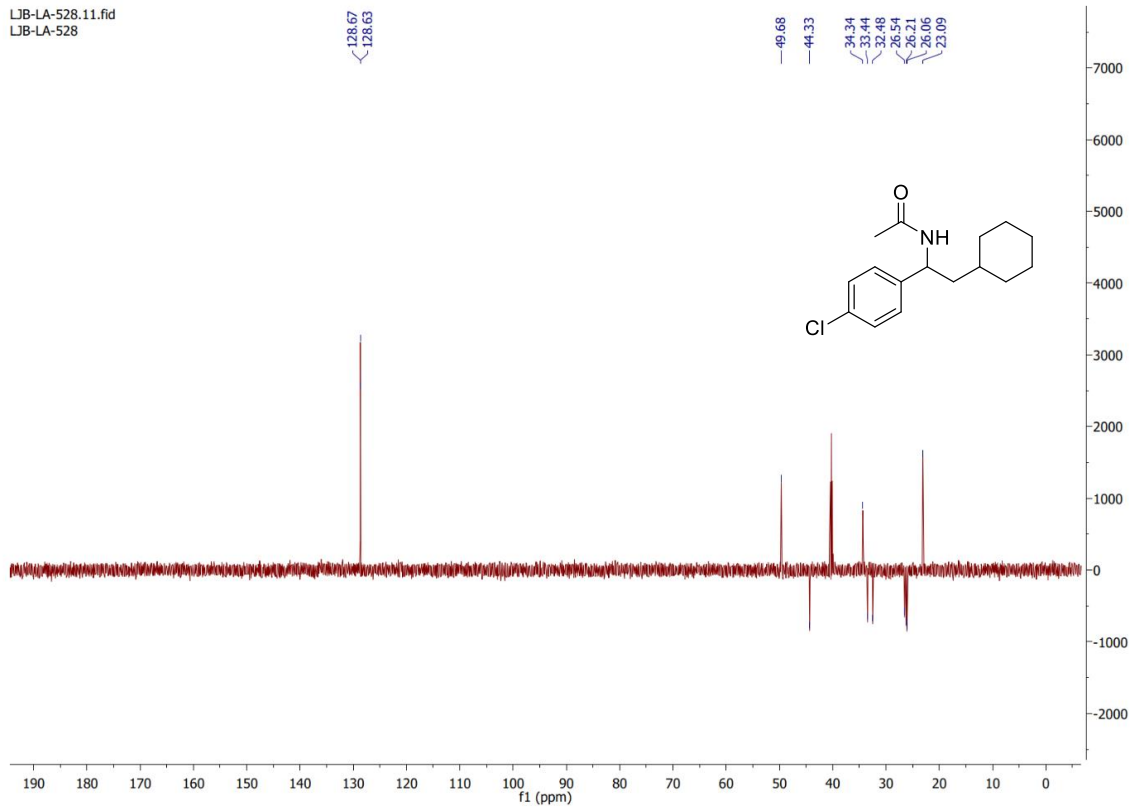
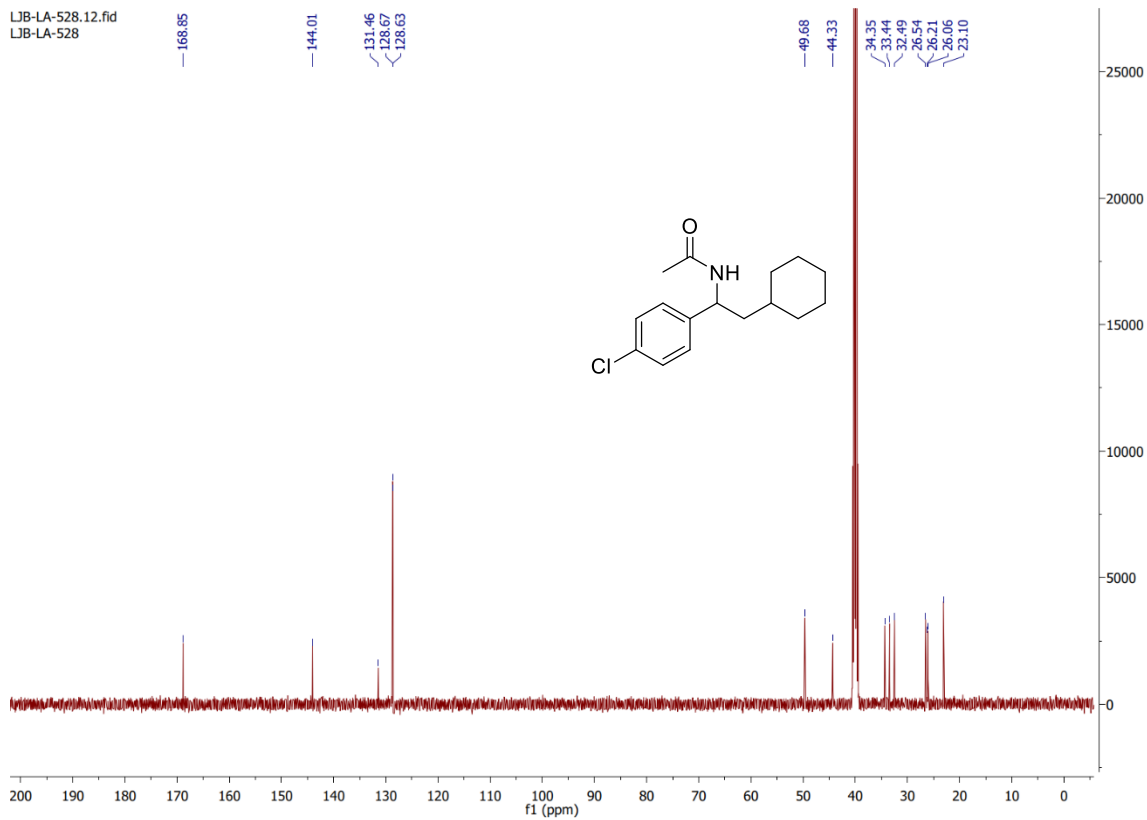


LJB-LA-526.106
LJB-LA-526

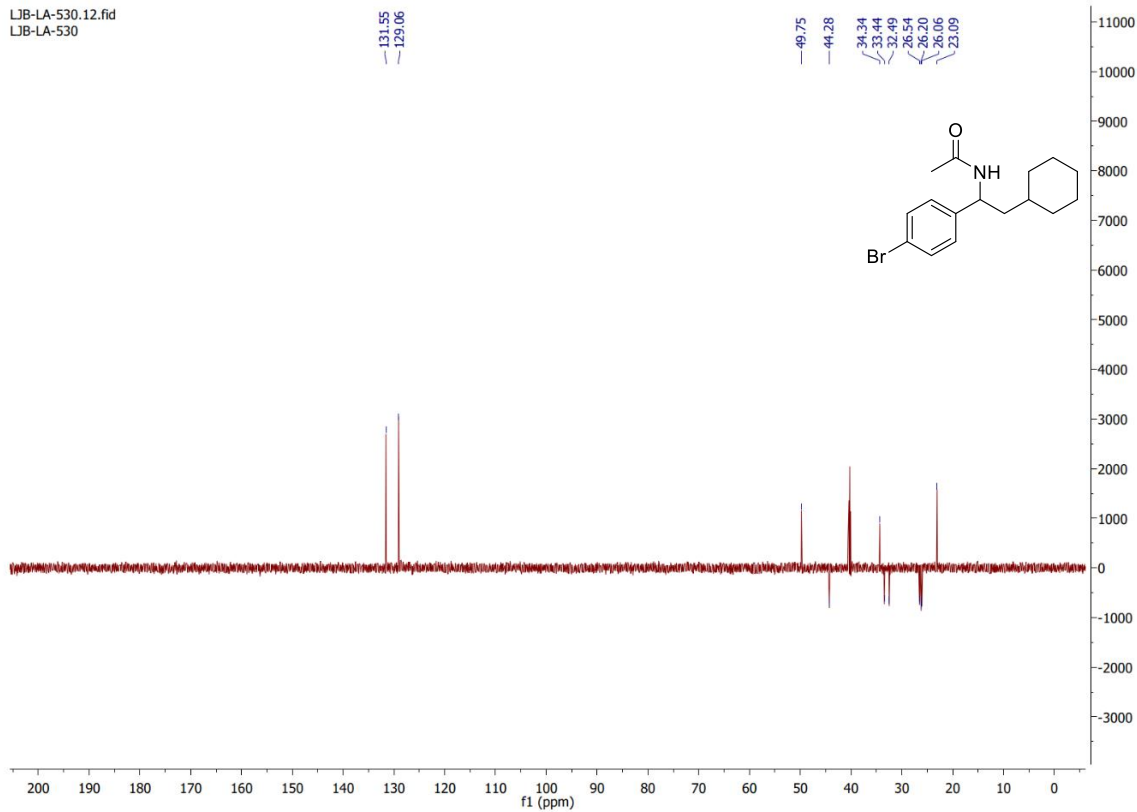




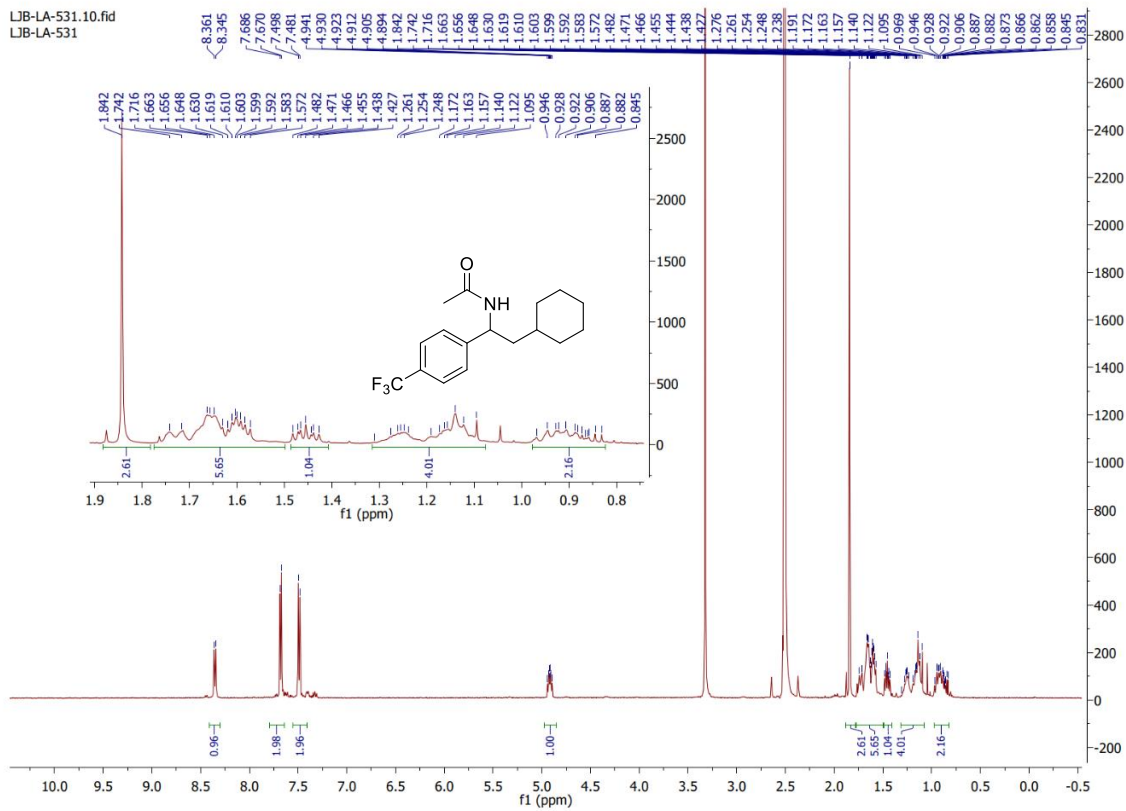




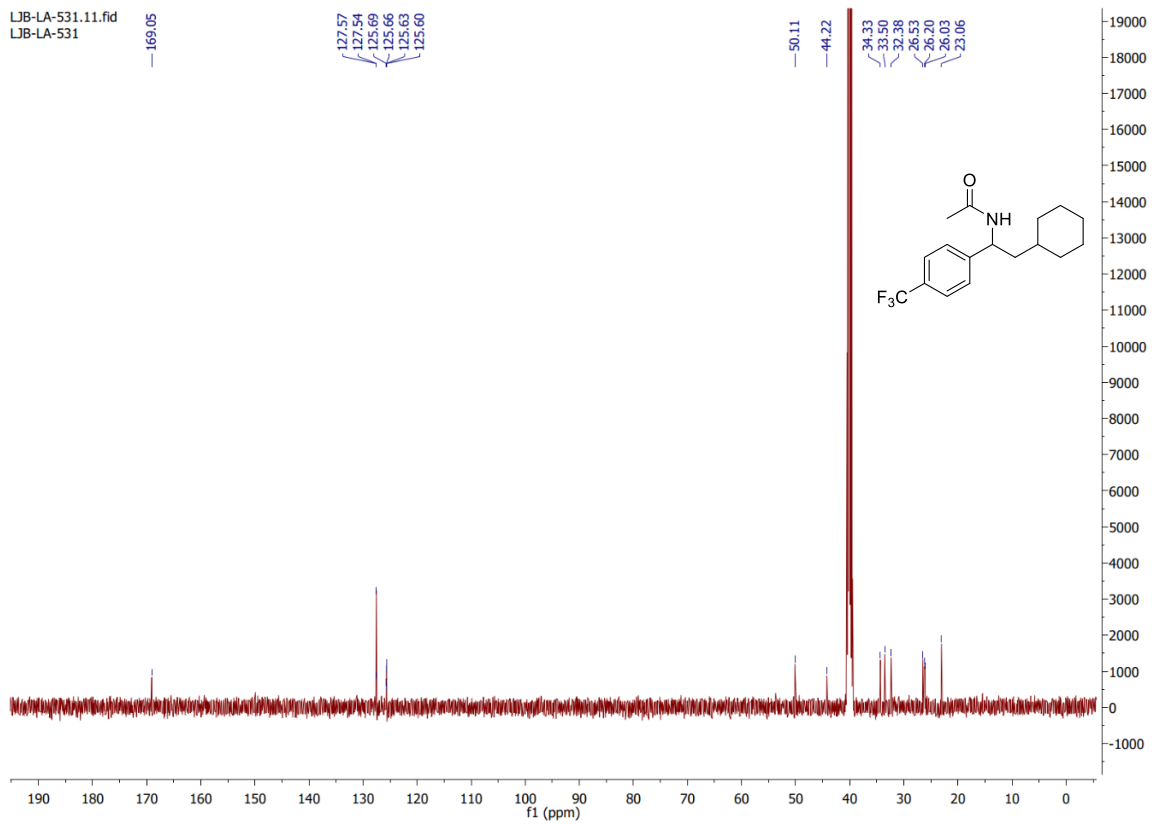
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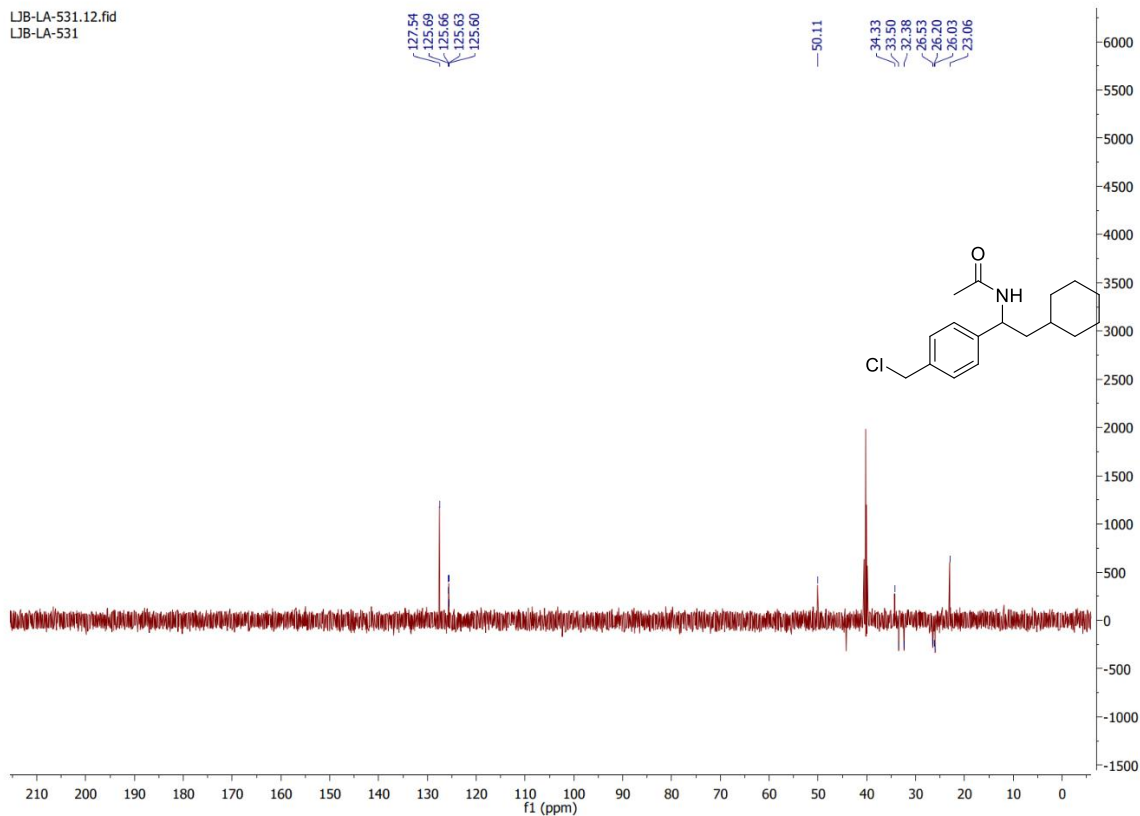
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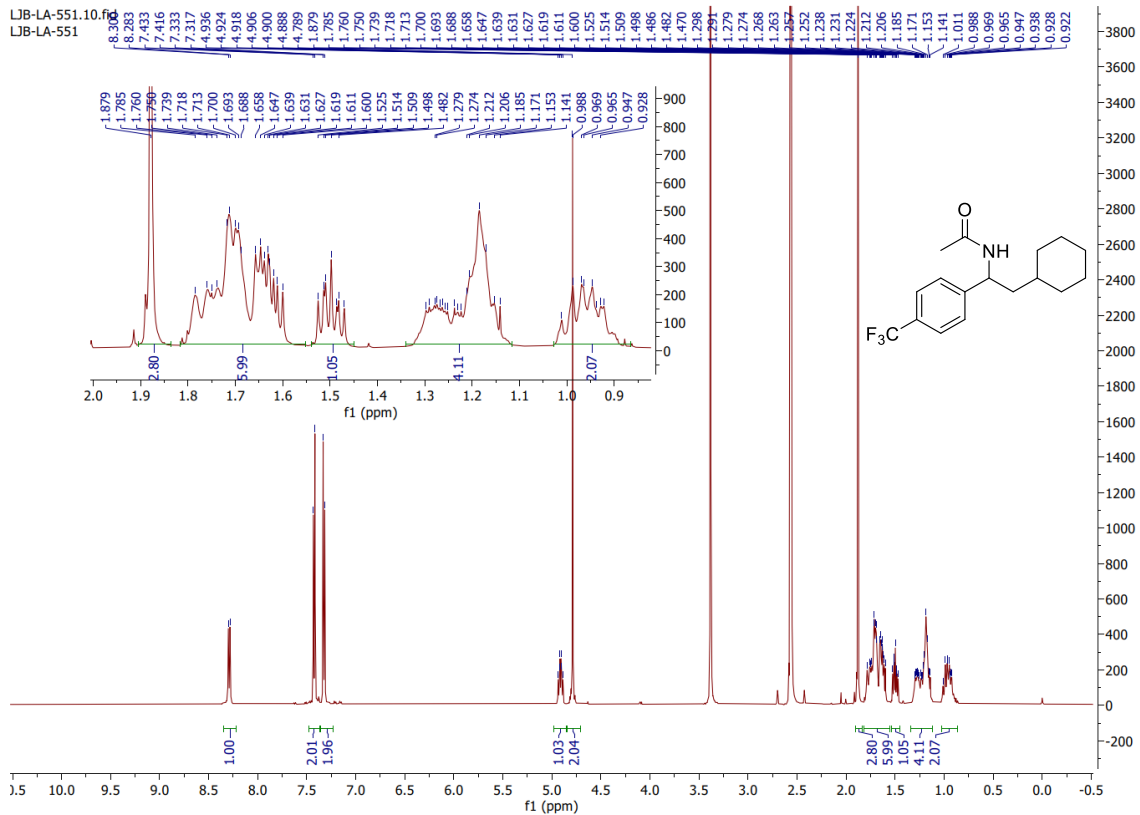
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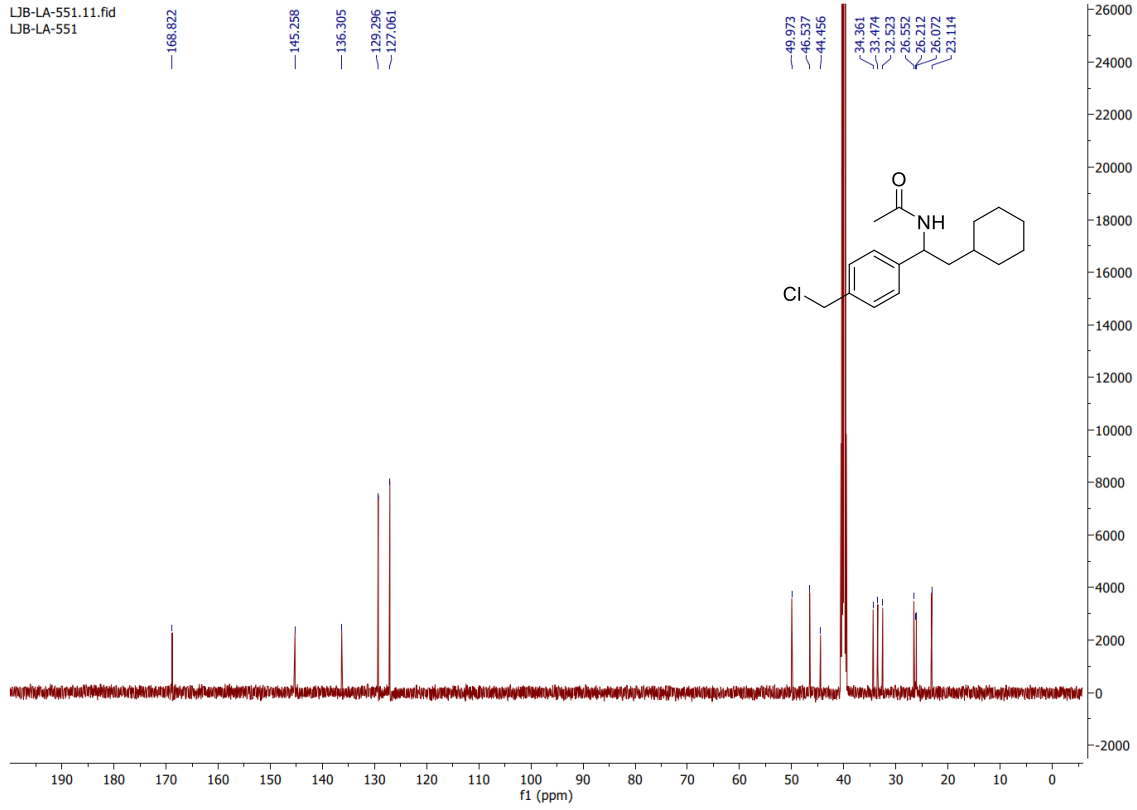
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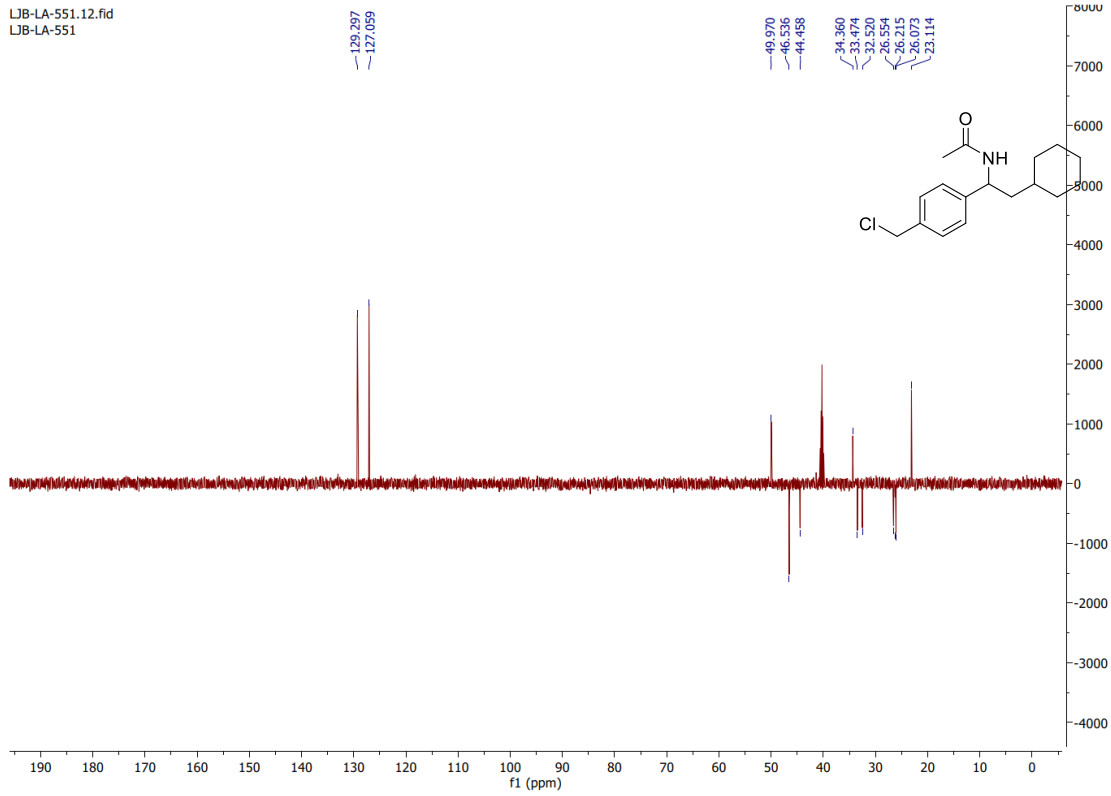
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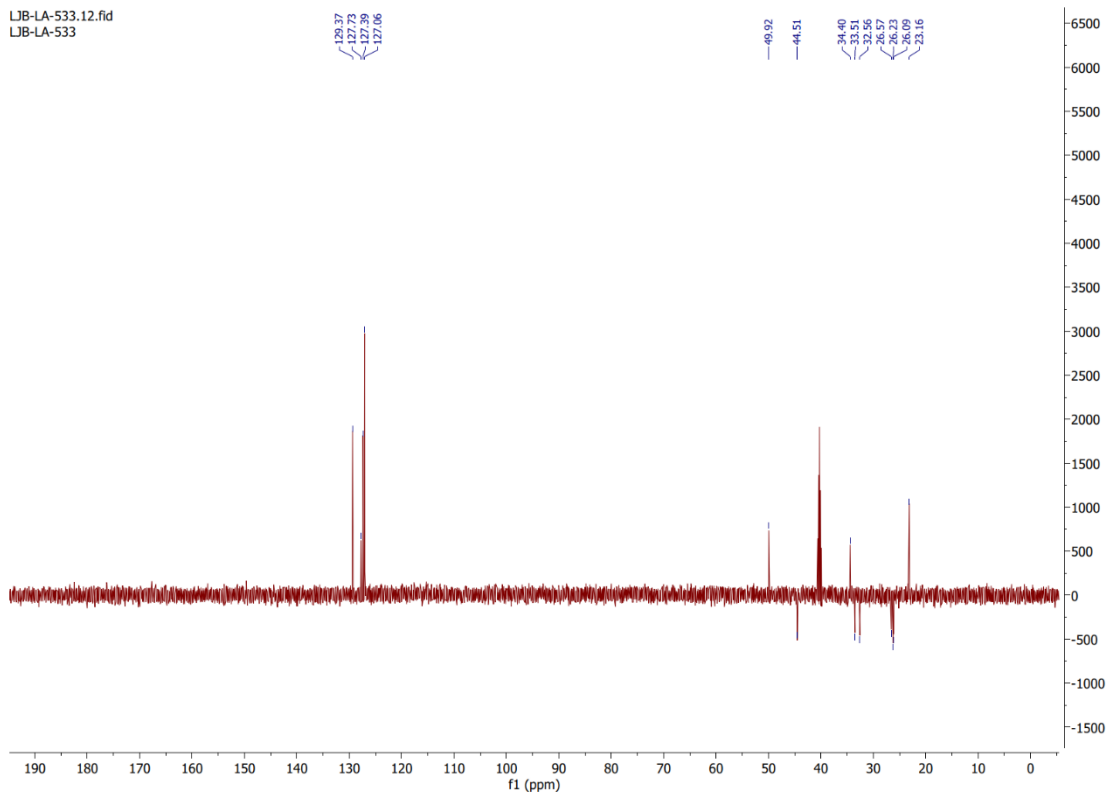
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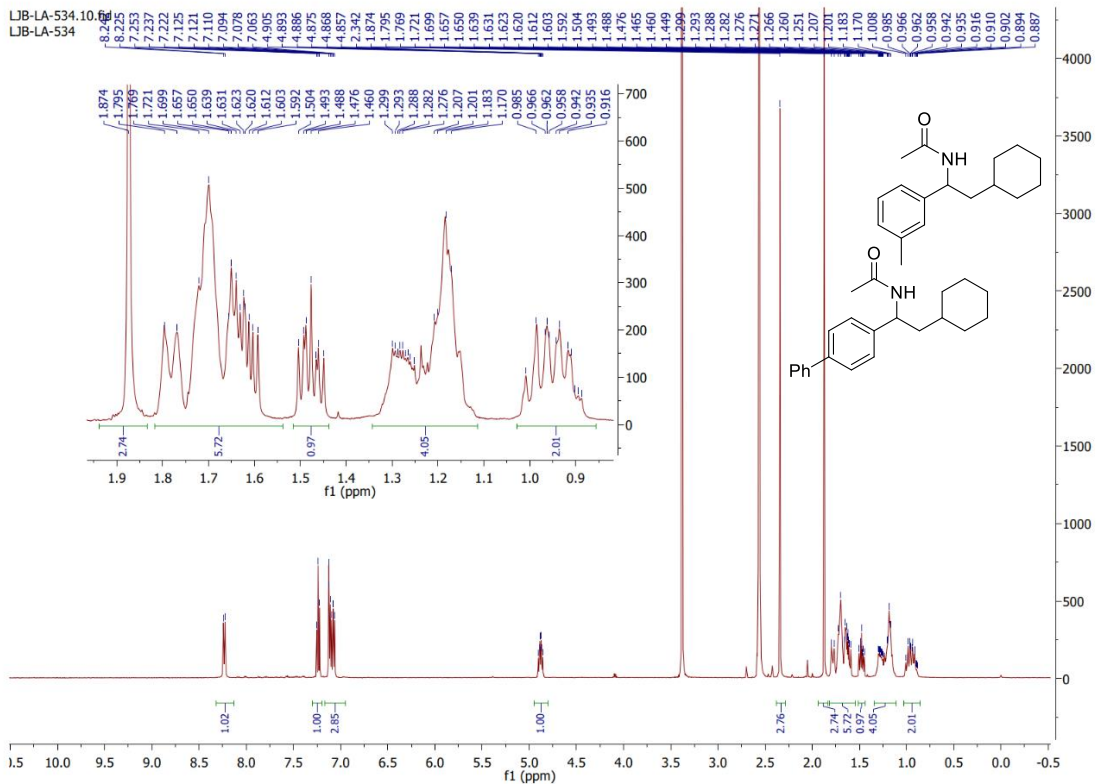
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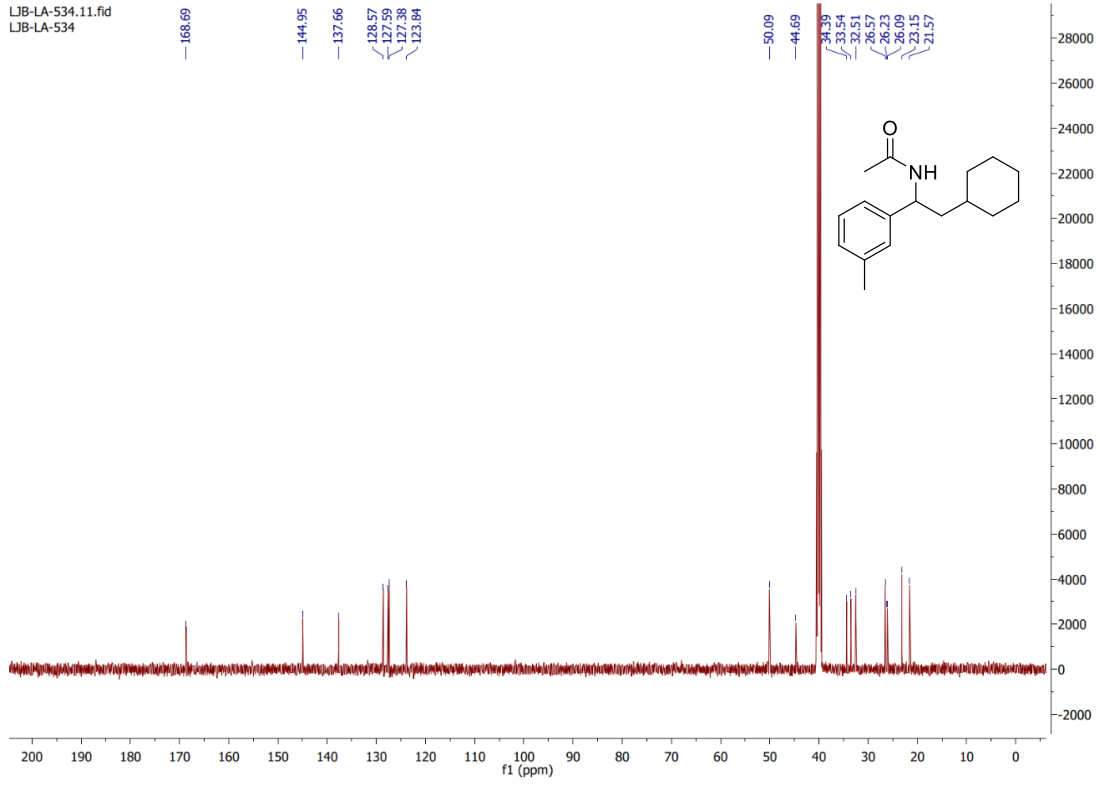
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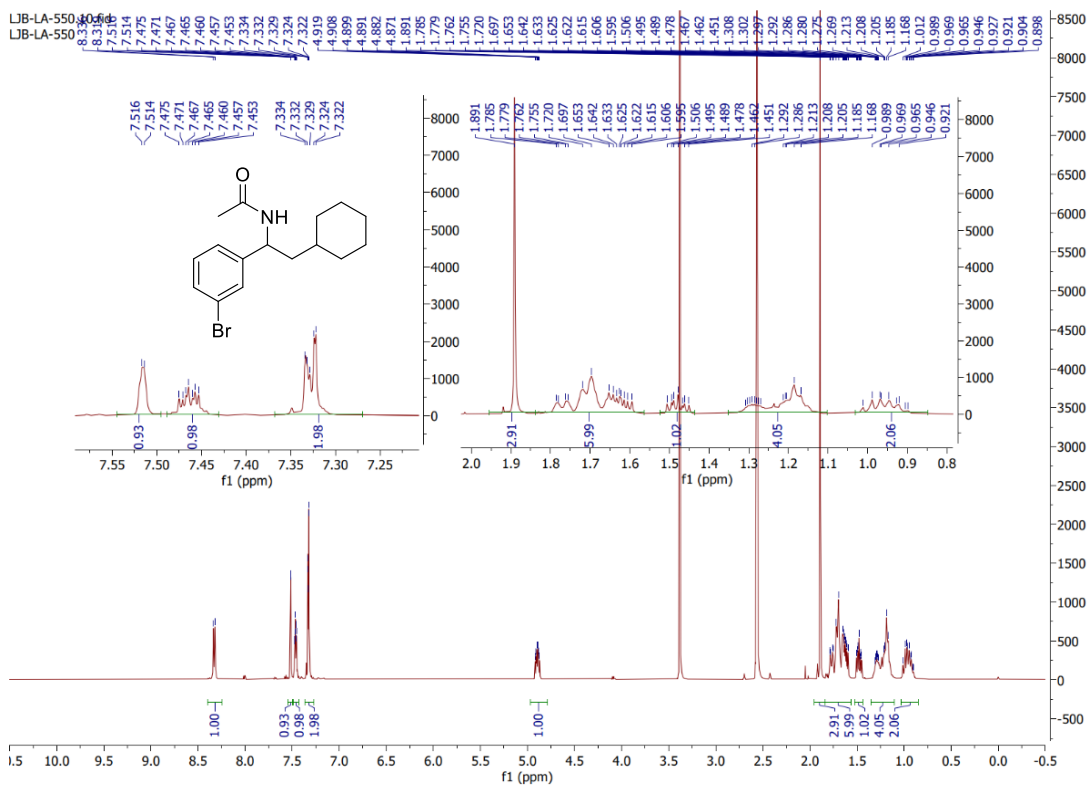
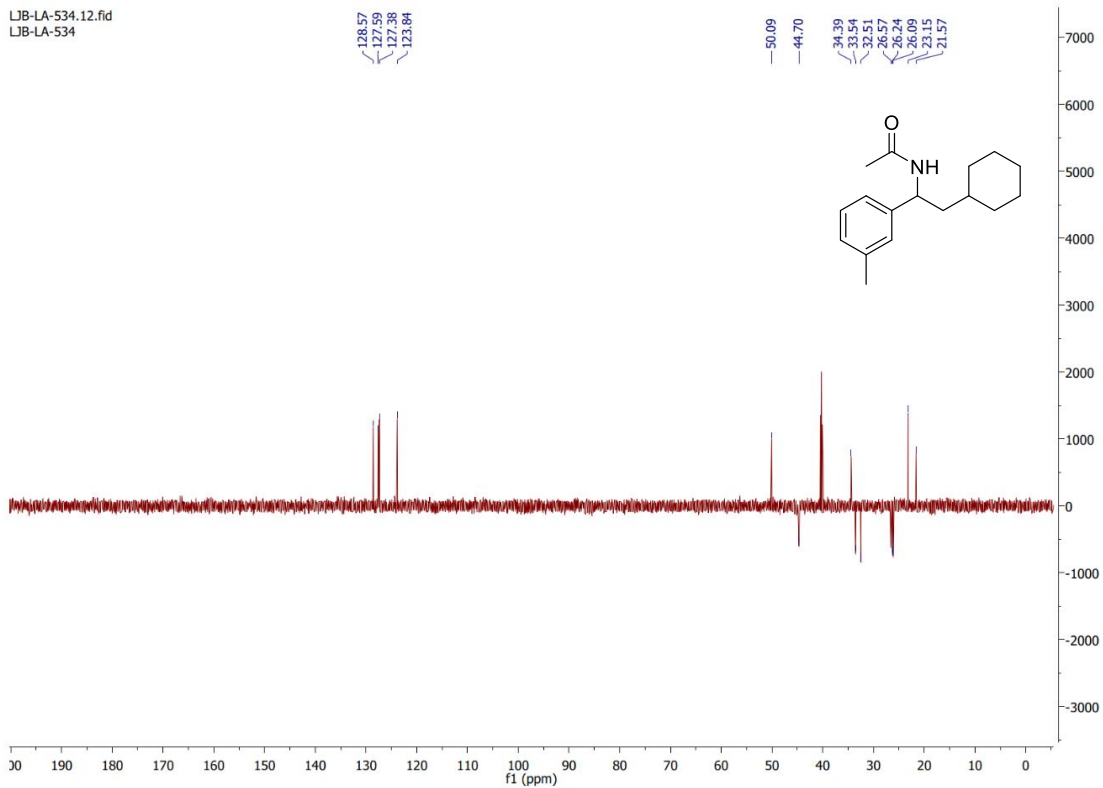
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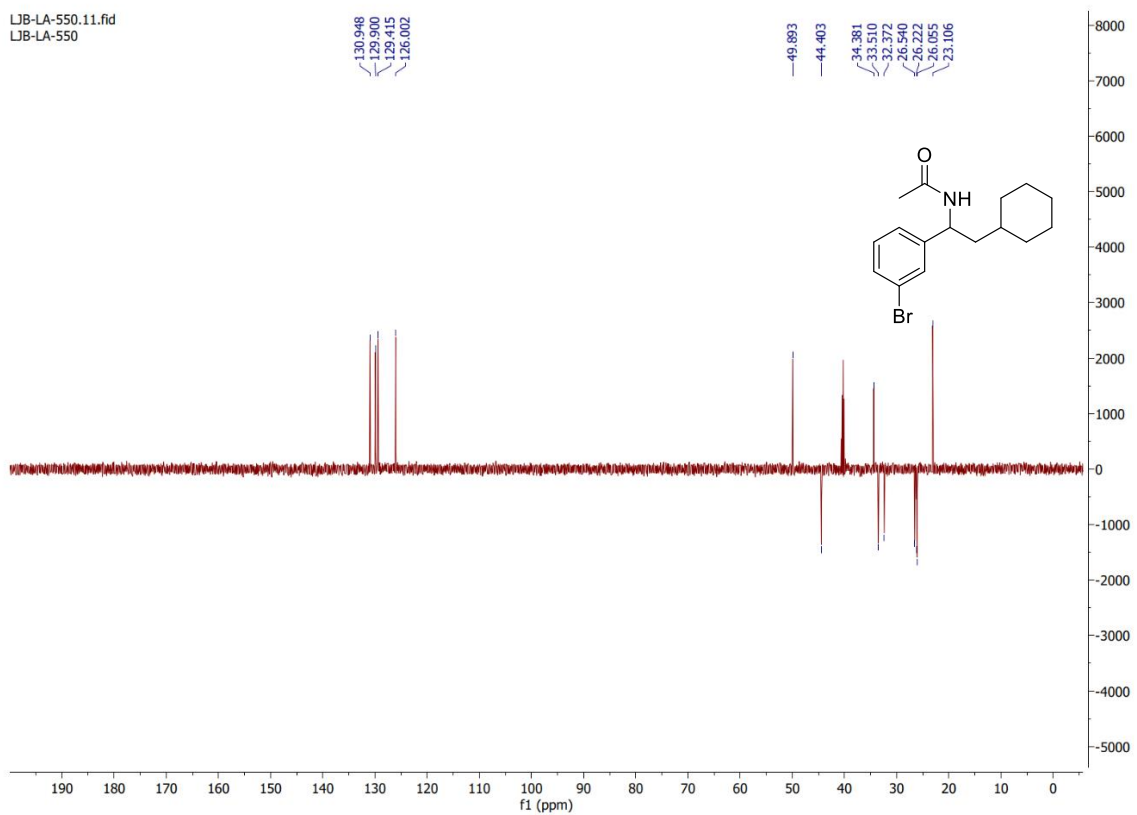
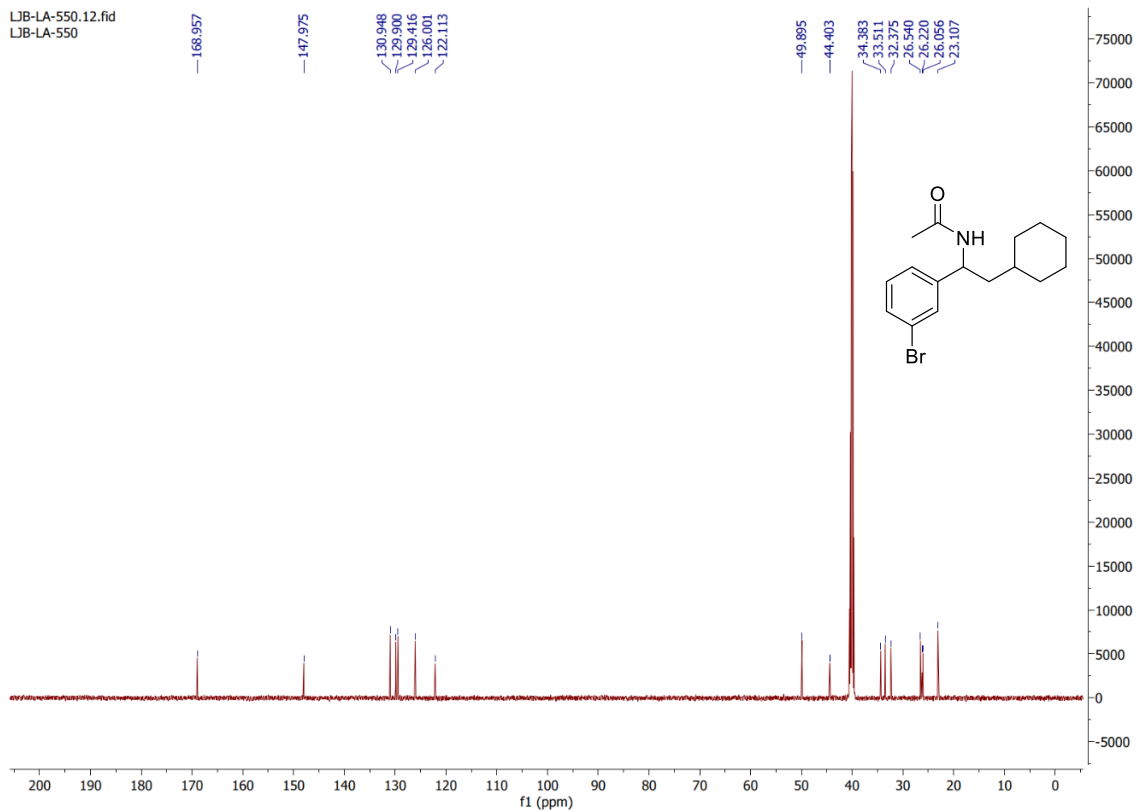


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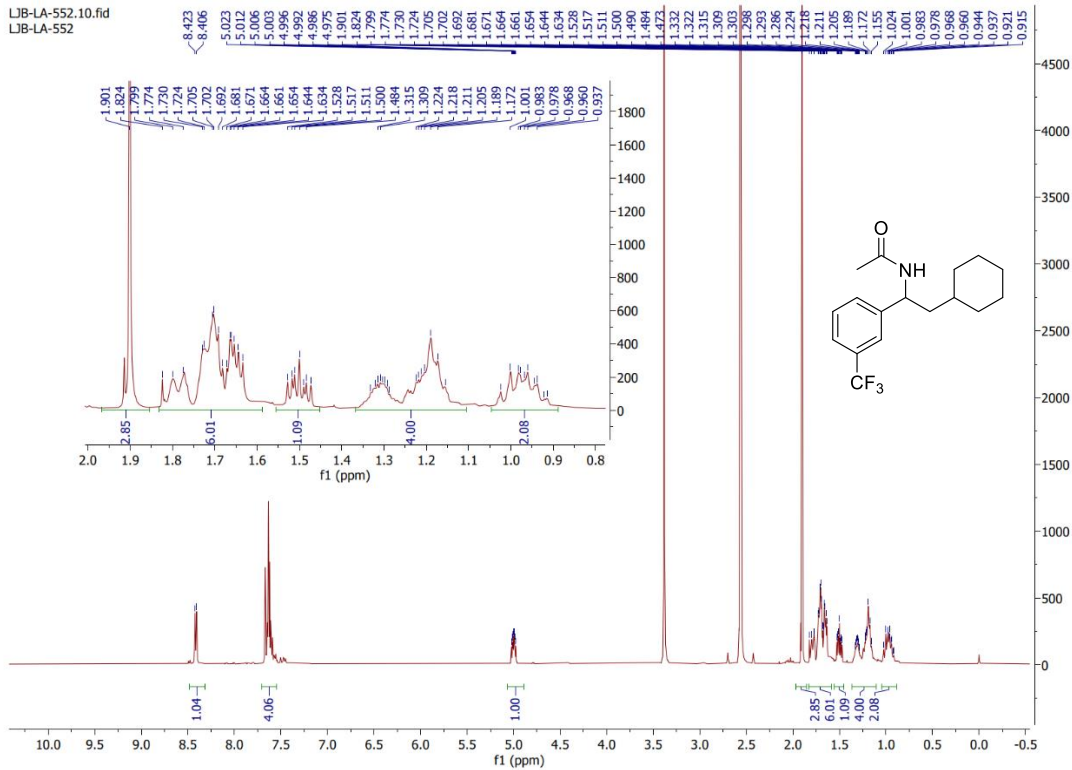


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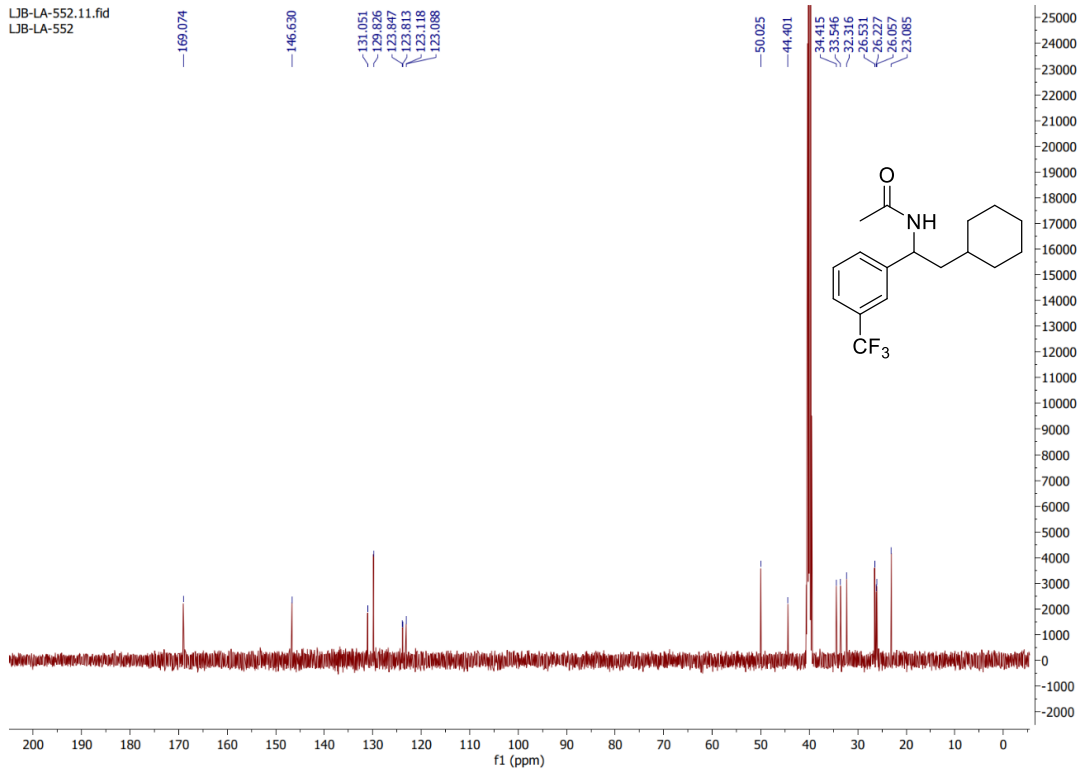




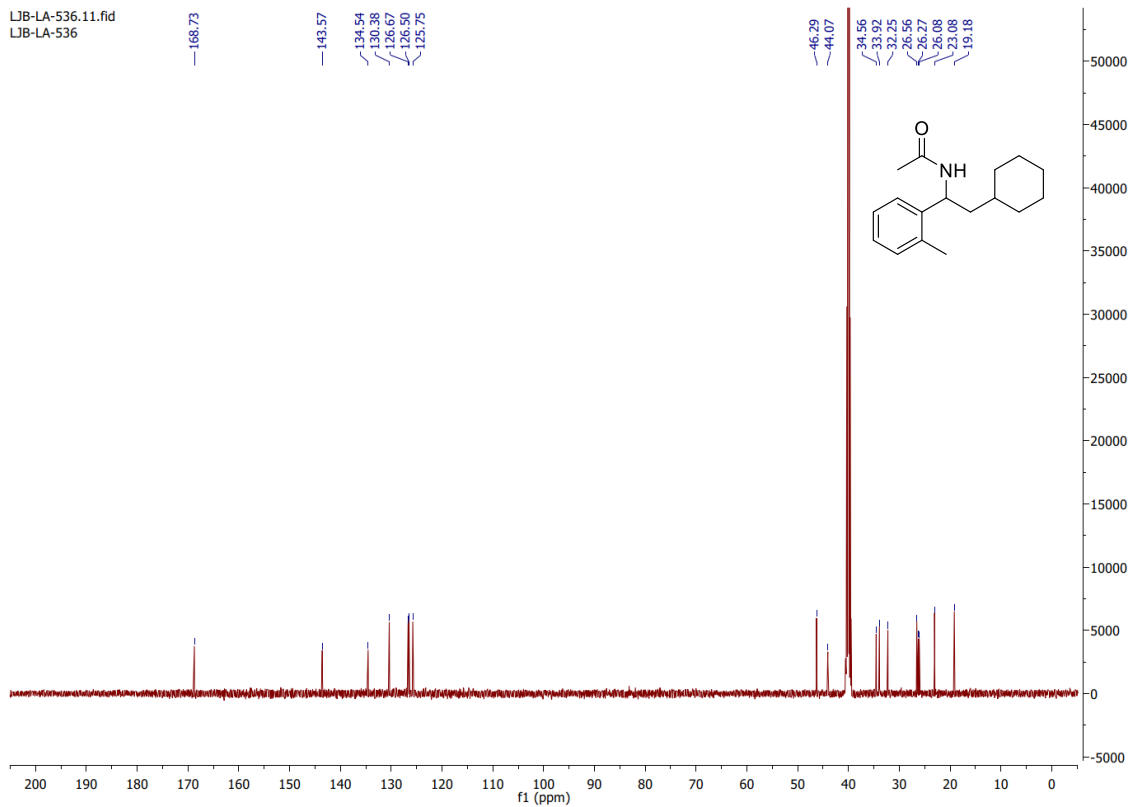
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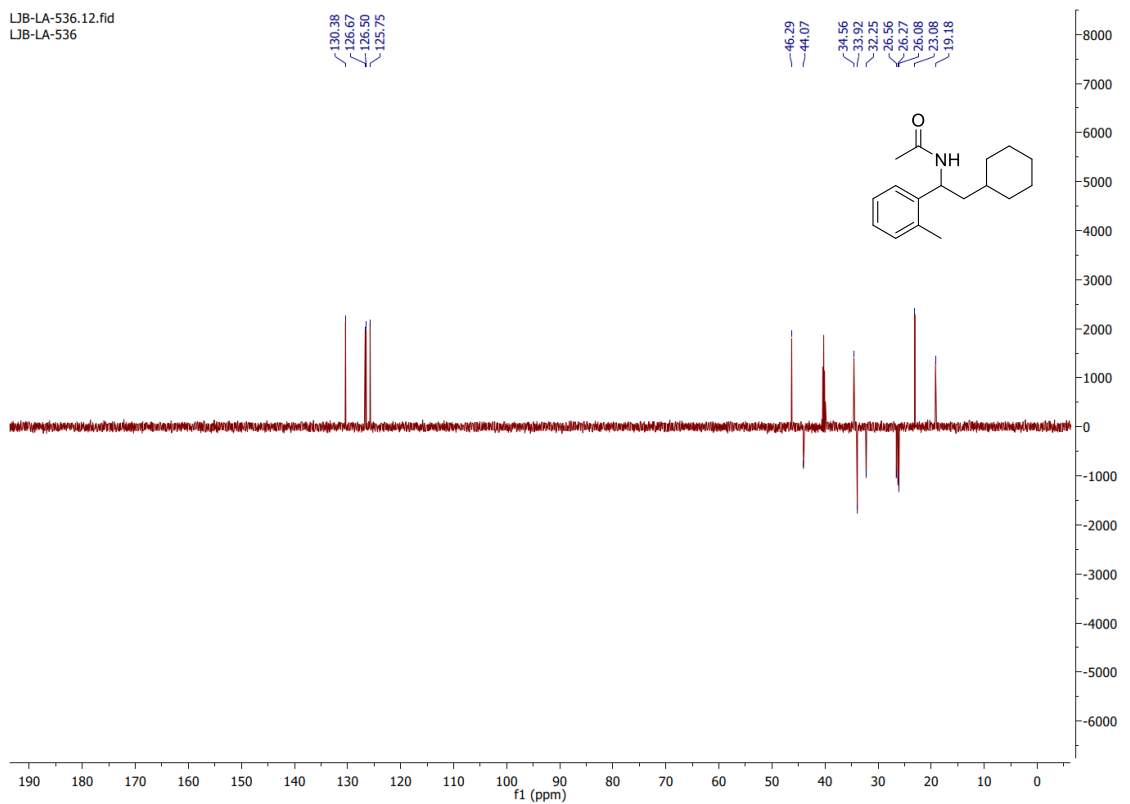
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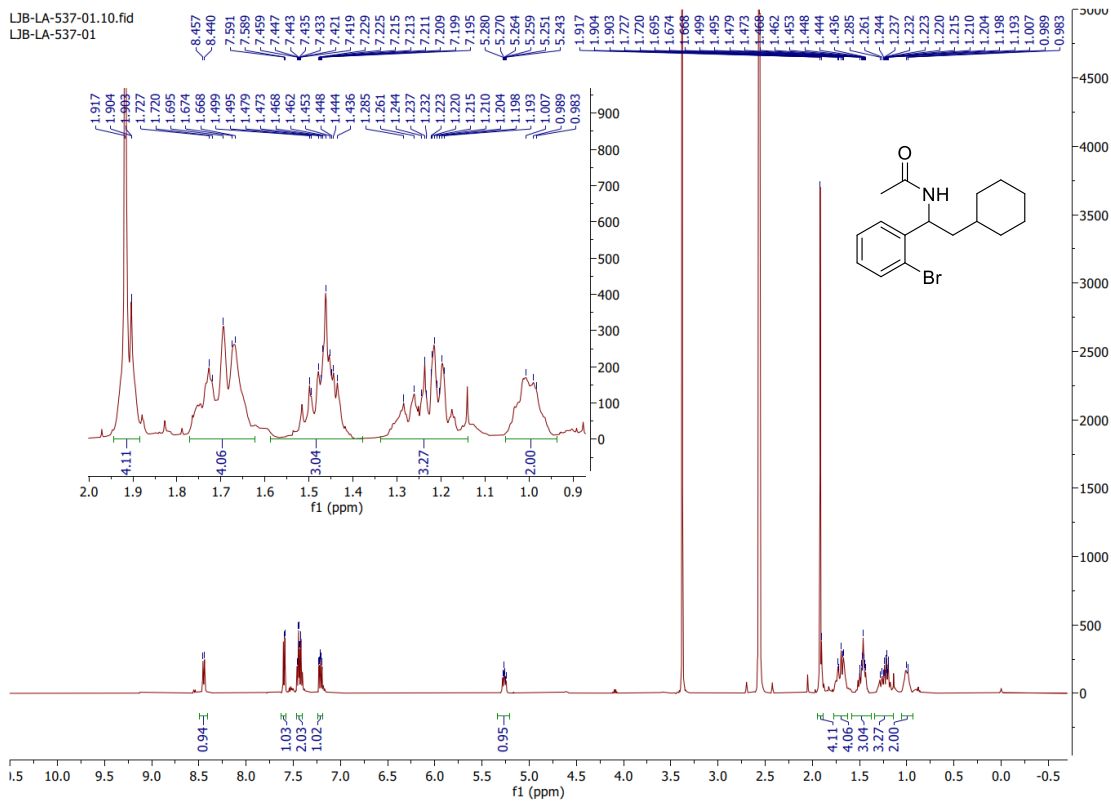
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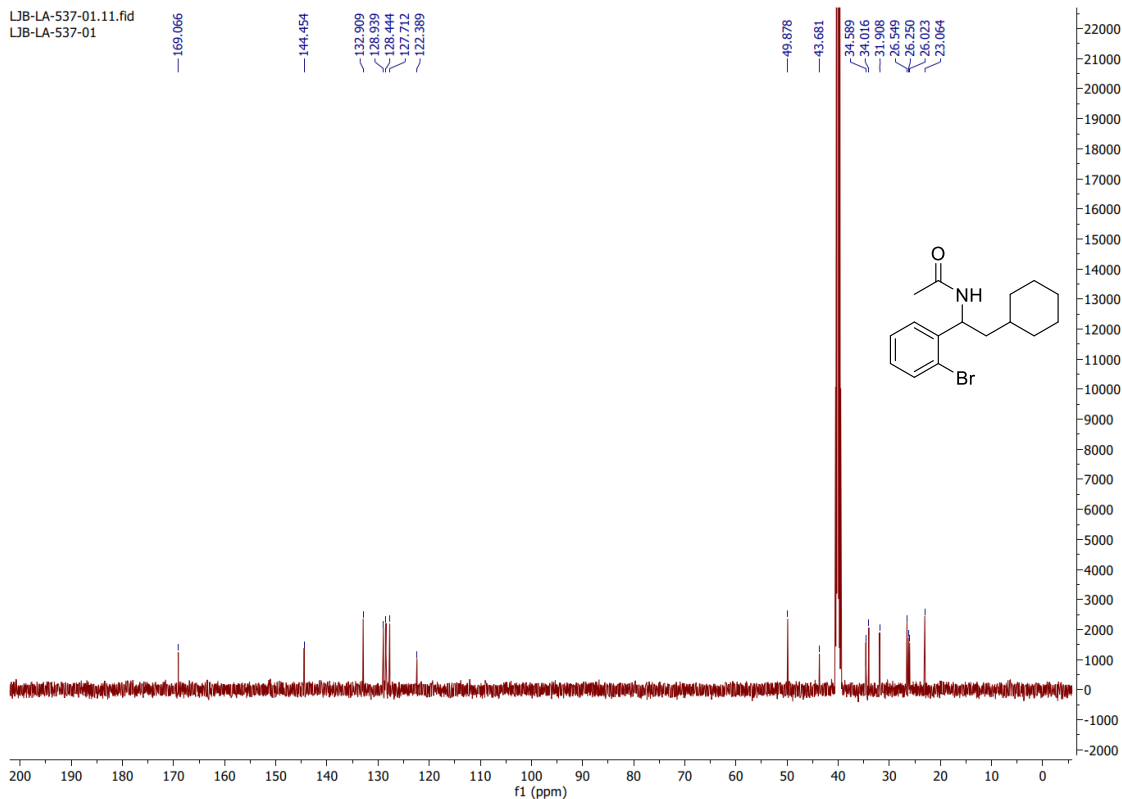
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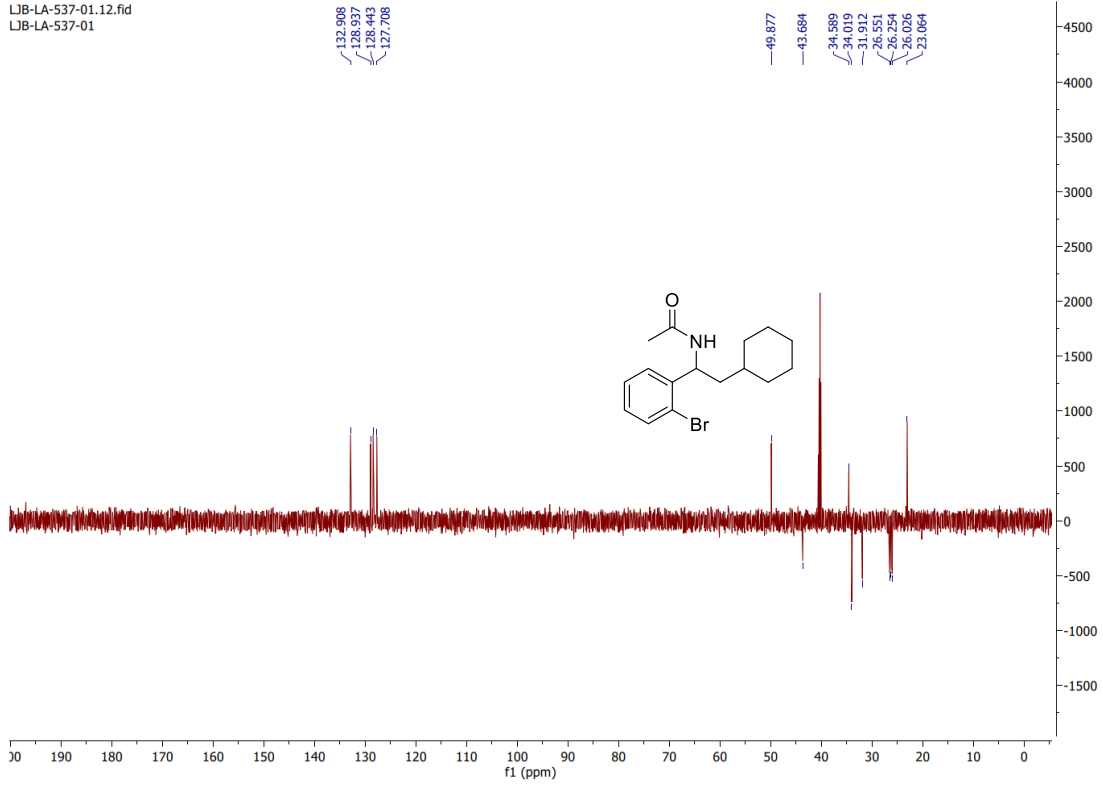
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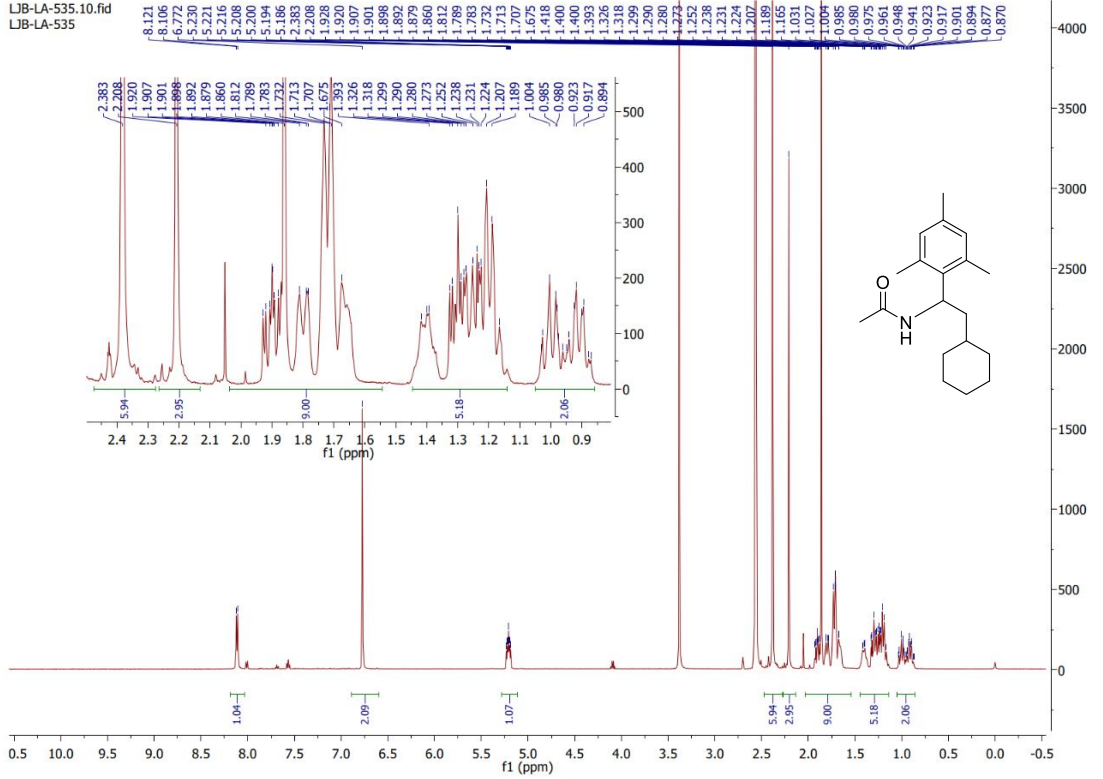
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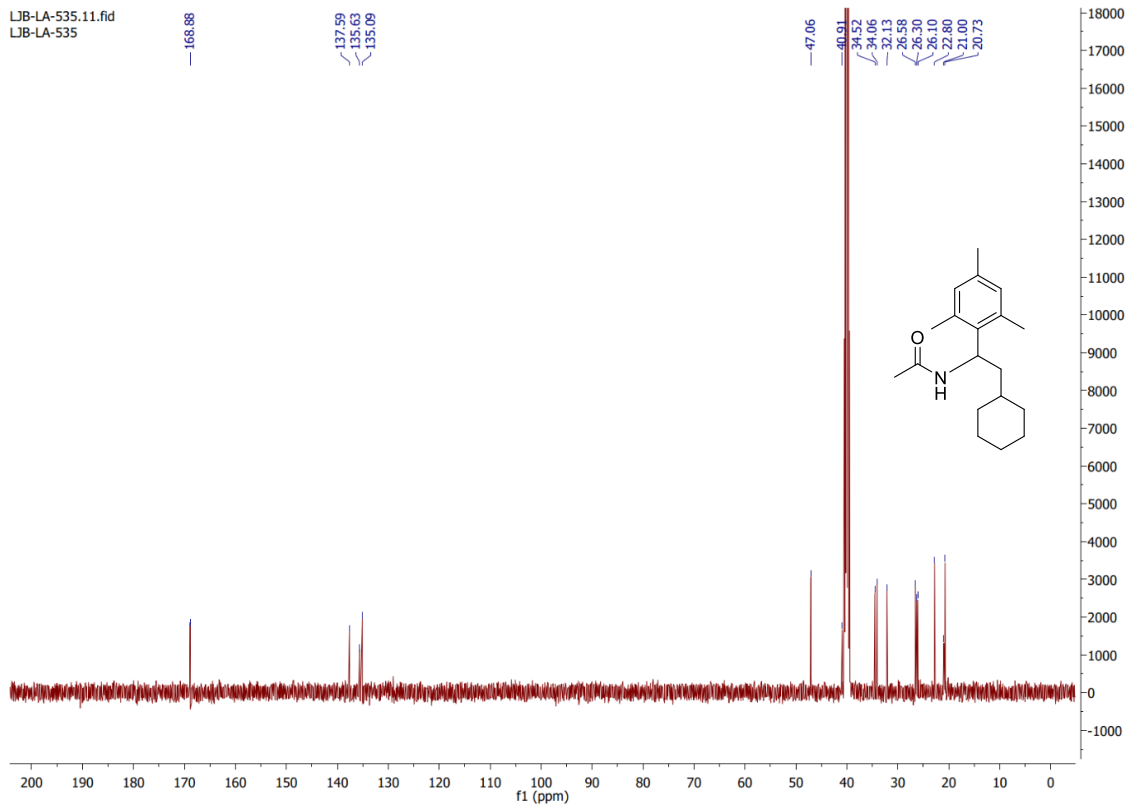
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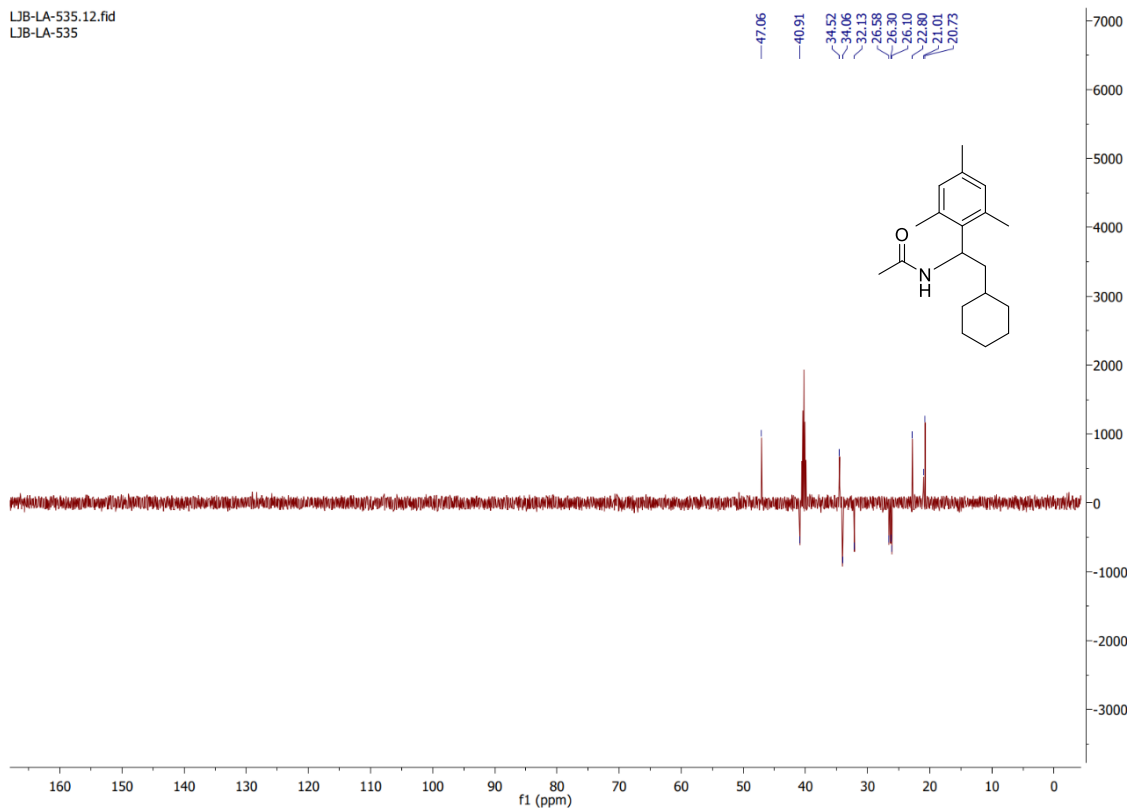
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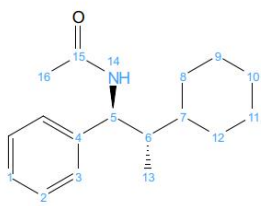


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LJB-LA-535.12.fid
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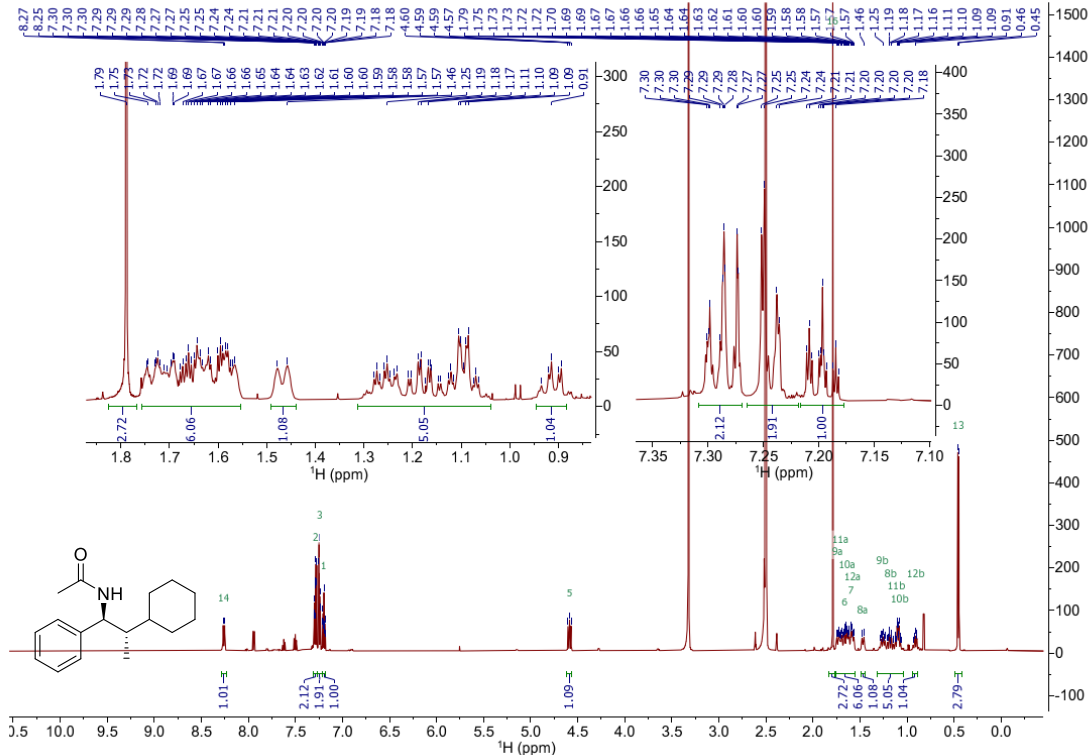
The NMR-data support a *rel*-S,S configuration.

See for comparison: LJB-LA-556-02

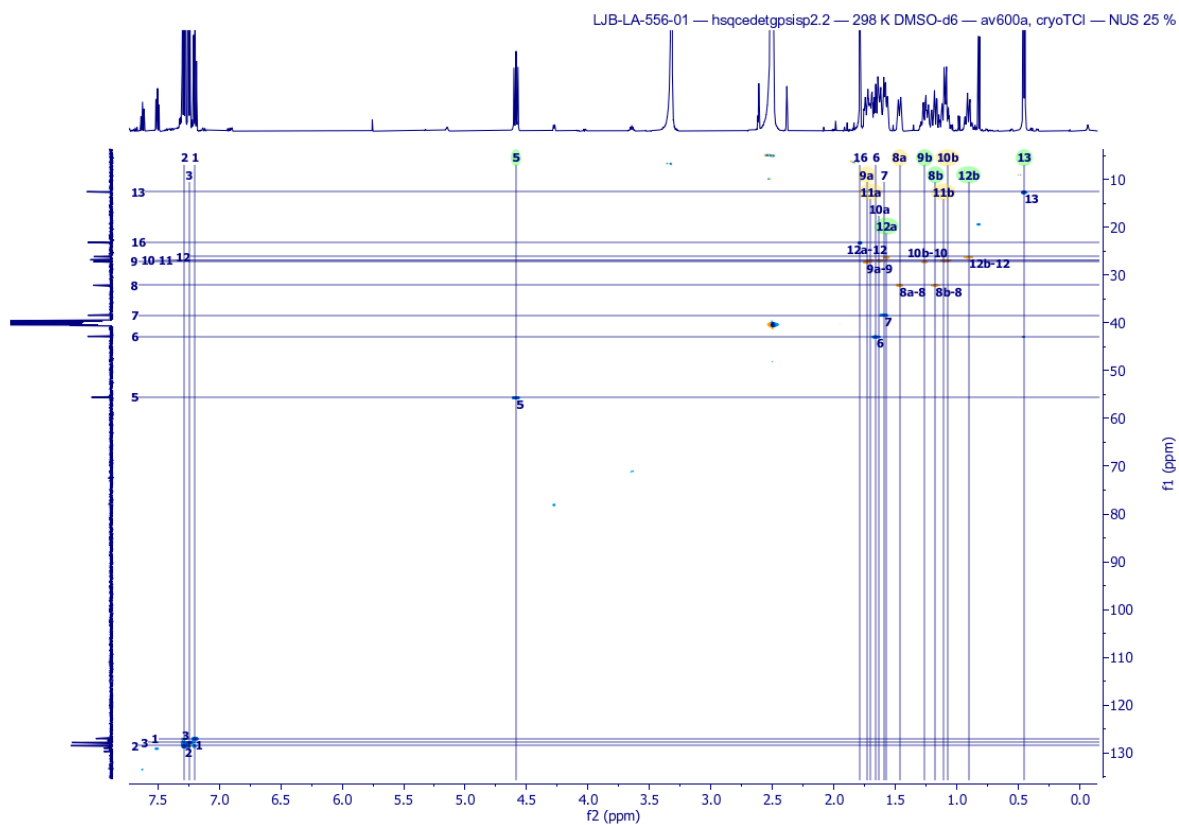
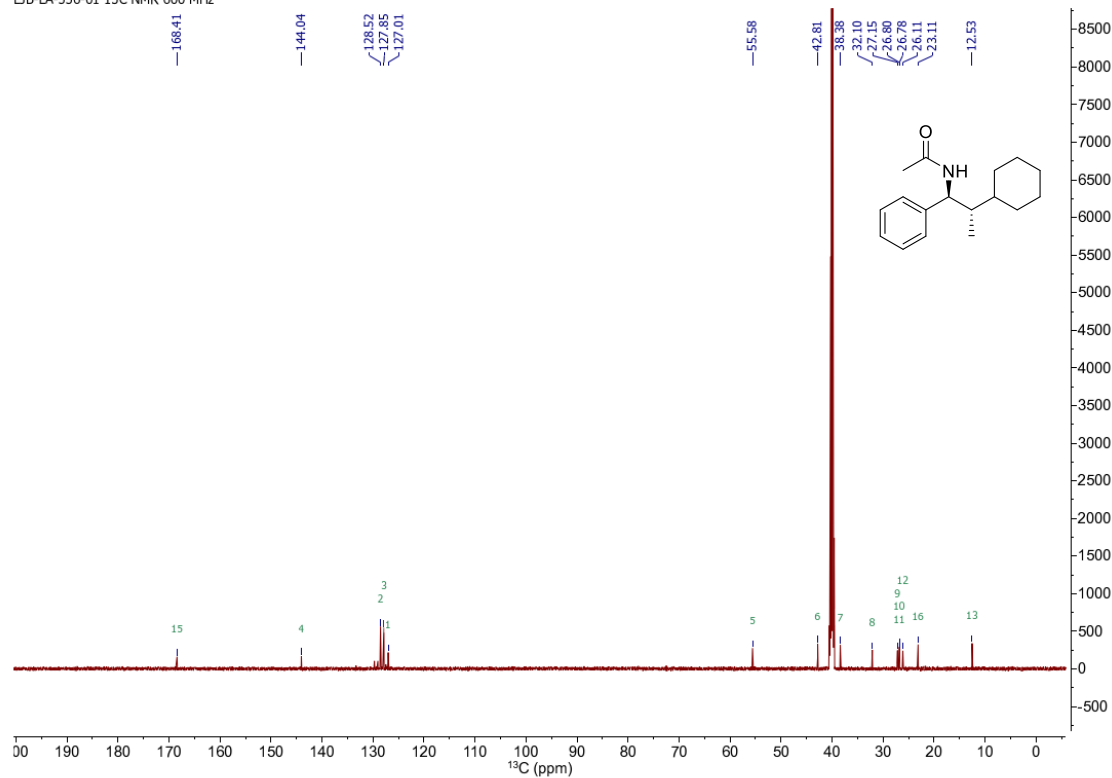
LJB-LA-556-01
DMSO-d₆; 298 K; 2 mg; AV600a

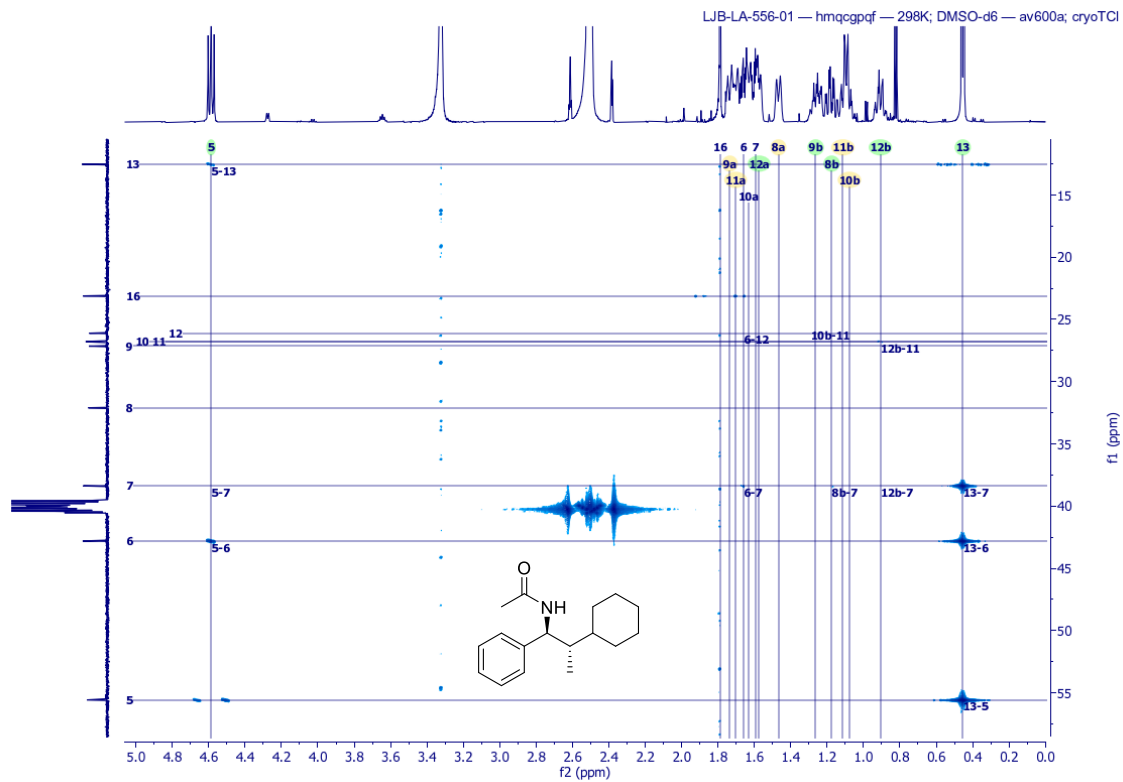
| Atom | δ (ppm) | J | HSQC | HMQC | COSY | NOESY | ¹ H-HMBC |
|------|---------|--|----------|--------------------|----------------------|---------------------|---------------------|
| C1 | 127.01 | | 1 | 3 | | | |
| H1 | 7.197 | m | 1 | 3 | 2, 3 | | |
| C2 | 128.52 | | 2 | 4 | | | |
| H2 | 7.285 | m | 2 | 4 | 1, 3 | | |
| C3 | 127.86 | | 3 | 1, 5 | | | |
| H3 | 7.244 | m | 3 | 1, 5 | 1, 2 | 5, 6, 13, 14 | |
| C4 | 144.04 | | | 2, 5 | | | |
| C5 | 55.58 | | 5 | 3, 13, 14 | | | |
| H5 | 4.587 | d 10.60(6), d 9.20(14) | 5 | 3, 4, 6, 7, 13, 15 | 6, 13, 14 | 3, 6, 12a, 13, 14 | |
| C6 | 42.81 | | 6 | 5, 13 | | | |
| H6 | 1.661 | m | 6 | 7, 12 | 5, 7, 13 | 3, 5, 8a, 13, 14 | |
| C7 | 38.38 | | 7 | 5, 6, 8b, 12b, 13 | | | |
| H7 | 1.595 | m (ax) | 7 | | 6, 8a, 8b, 12b | 8a, 9b, 13, 14 | |
| C8 | 32.10 | | 8a, 8b | | | | |
| H8a | 1.468 | (eq); dm 12.40(8b) | 8 | | 7, 8b, 9a, 9b, 12a | 6, 7, 8b, 9a, 9b | |
| H8b | 1.175 | (ax); d 12.40(7), d 12.4(8a), d 12.10(9b), d 3.20(9a) | 8 | 7, 12 | 7, 8a, 9a, 9b | 8a, 9a, 12b, 13 | |
| C9 | 27.15 | | 9a, 9b | | | | |
| H9a | 1.735 | (eq); dm 12.50(9b) | 9 | | 8a, 8b, 9b, 10a, 10b | 8a, 8b, 9b, 10a | |
| H9b | 1.263 | (ax); d 12.5(9a), d 12.1(10b); d 12.1(8b); t 3.50(8a, 10a) | 9 | 11 | 8a, 8b, 9a, 10a, 10b | 7, 8a, 9a, 10a | |
| C10 | 26.80 | | 10a, 10b | | | | |
| H10a | 1.532 | m (eq) | 10 | | 9a, 9b, 10b | 9a, 9b, 10b, 11b | |
| H10b | 1.077 | m (ax) | 10 | 11 | 9a, 9b, 10a, 11b | 10a | |
| C11 | 26.78 | | 11a, 11b | 9b, 10b, 12b | | | |
| H11a | 1.703 | m (eq) | 11 | | 11b, 12a, 12b | 11b, 12a, 12b | |
| H11b | 1.113 | m (ax) | 11 | | 10b, 11a, 12a, 12b | 10a, 11a | |
| C12 | 26.11 | | 12a, 12b | 6, 8b | | | |
| H12a | 1.574 | m (eq) | 12 | | 8a, 11a, 11b, 12b | 5, 11a, 12b | |
| H12b | 0.907 | (ax); q 12.10(7, 11b, 12a), d 3.50(11a) | 12 | 7, 11 | 7, 11a, 11b, 12a | 8b, 11a, 12a, 13 | |
| C13 | 12.54 | | 13 | 5 | | | |
| H13 | 0.454 | d 7.00(6) | 13 | 5, 6, 7 | 5, 6 | 3, 5, 6, 7, 8b, 12b | |
| H14 | -249.0 | | | | | | 14 |
| H14 | 8.260 | d 9.20(5) | | 5, 15 | 5 | 3, 5, 6, 7, 16 | 14 |
| C15 | 168.41 | | | 5, 14, 16 | | | |
| C16 | 23.11 | | 16 | | | | |
| H16 | 1.789 | s | 16 | 15 | | 14 | |

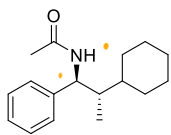
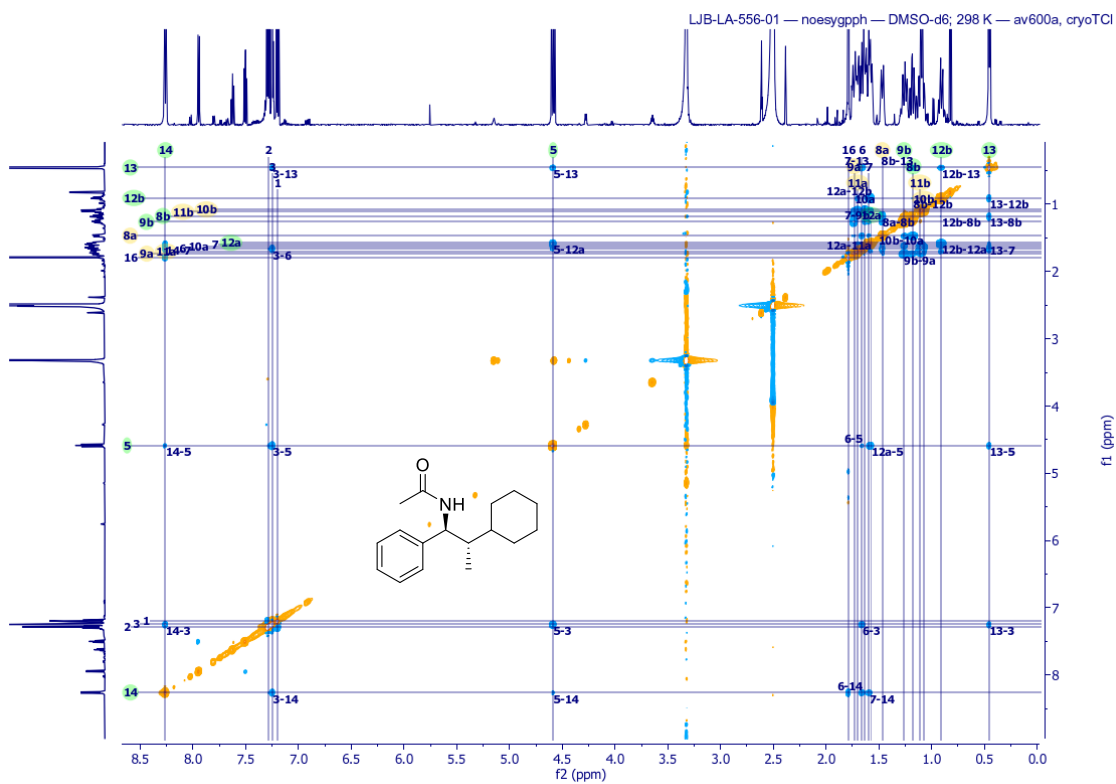
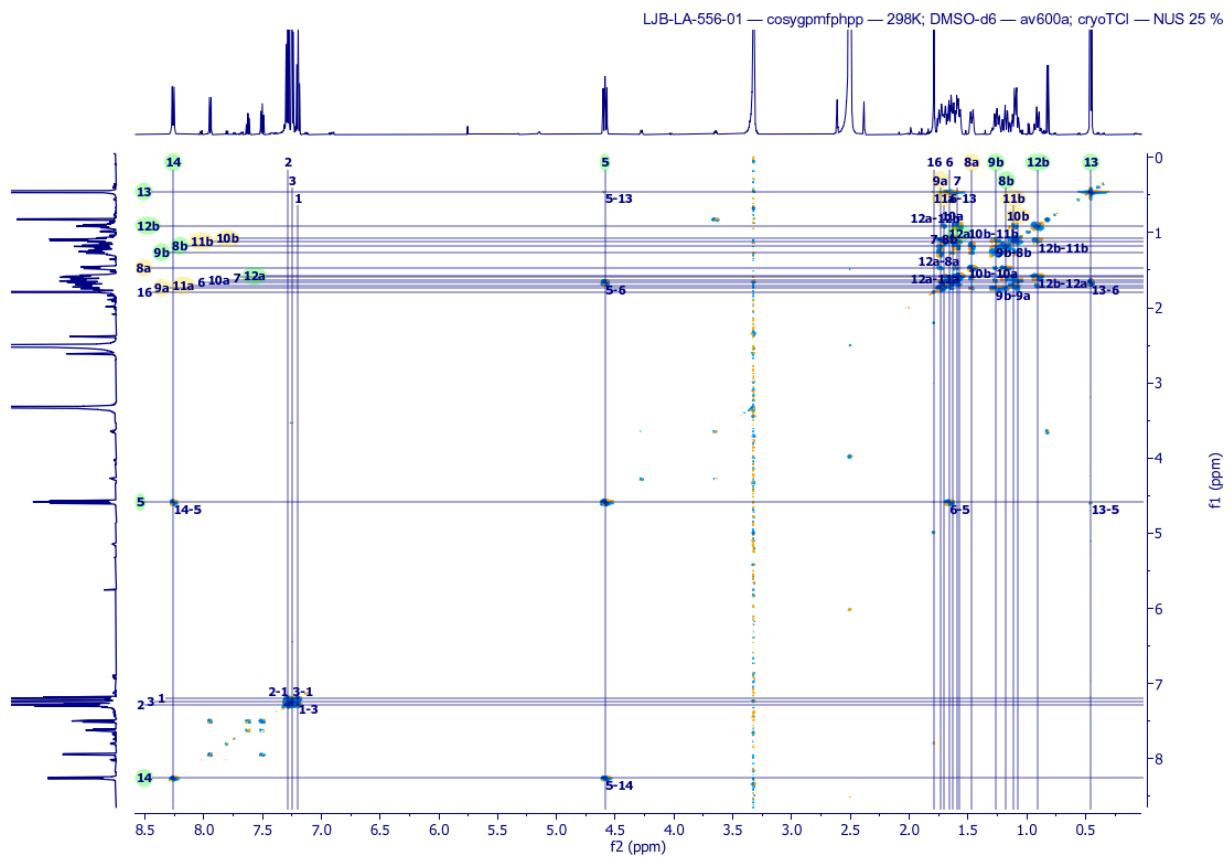
LJB-LA-556-01 ¹H NMR 600 MHz

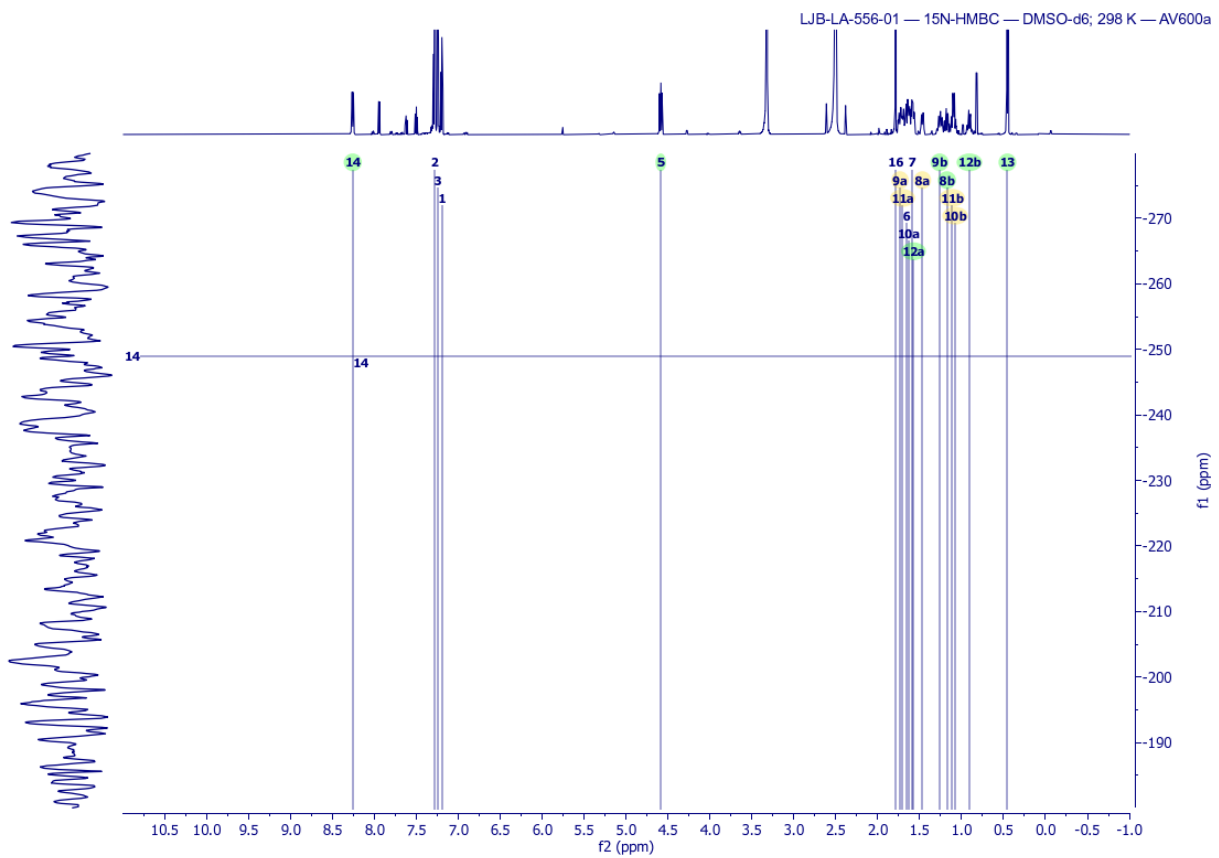


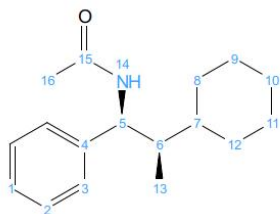
LJB-LA-556-01 13C NMR 600 MHz











LJB-LA-556-02
DMSO-d₆; 298 K; 2 mg; AV600a

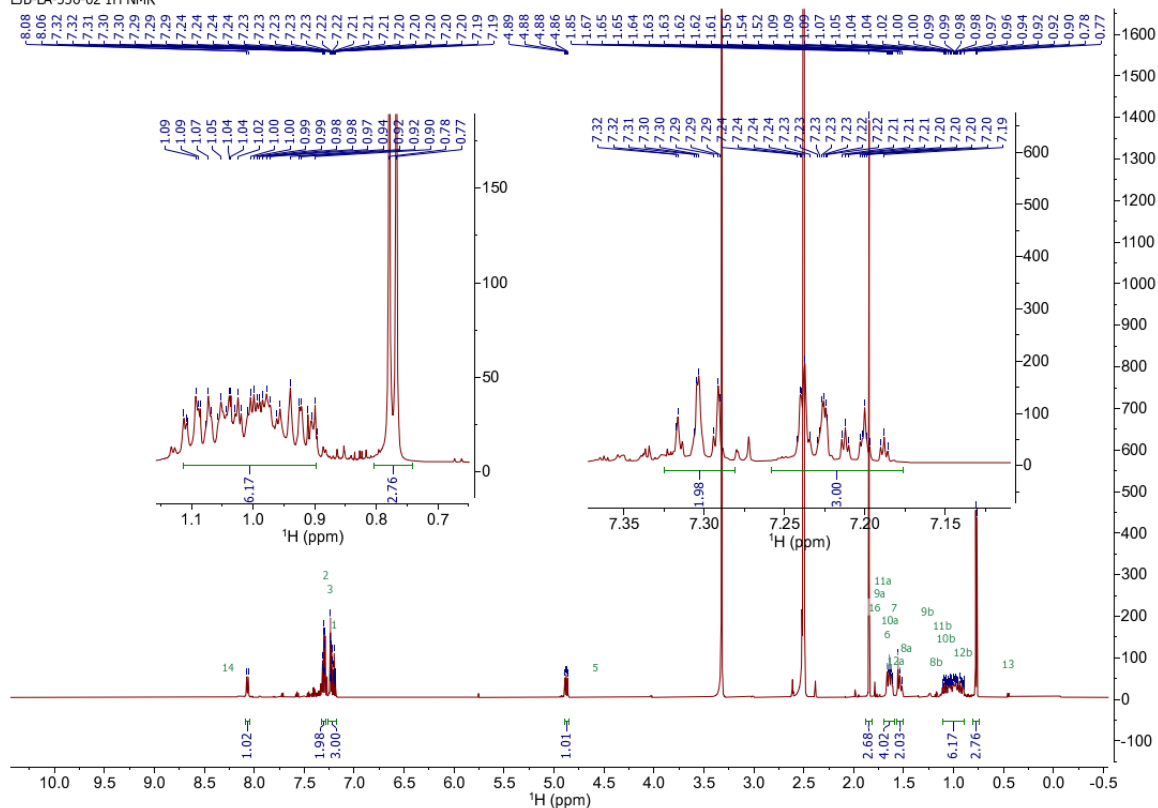
The NMR-data support a *rel*-S,R configuration.

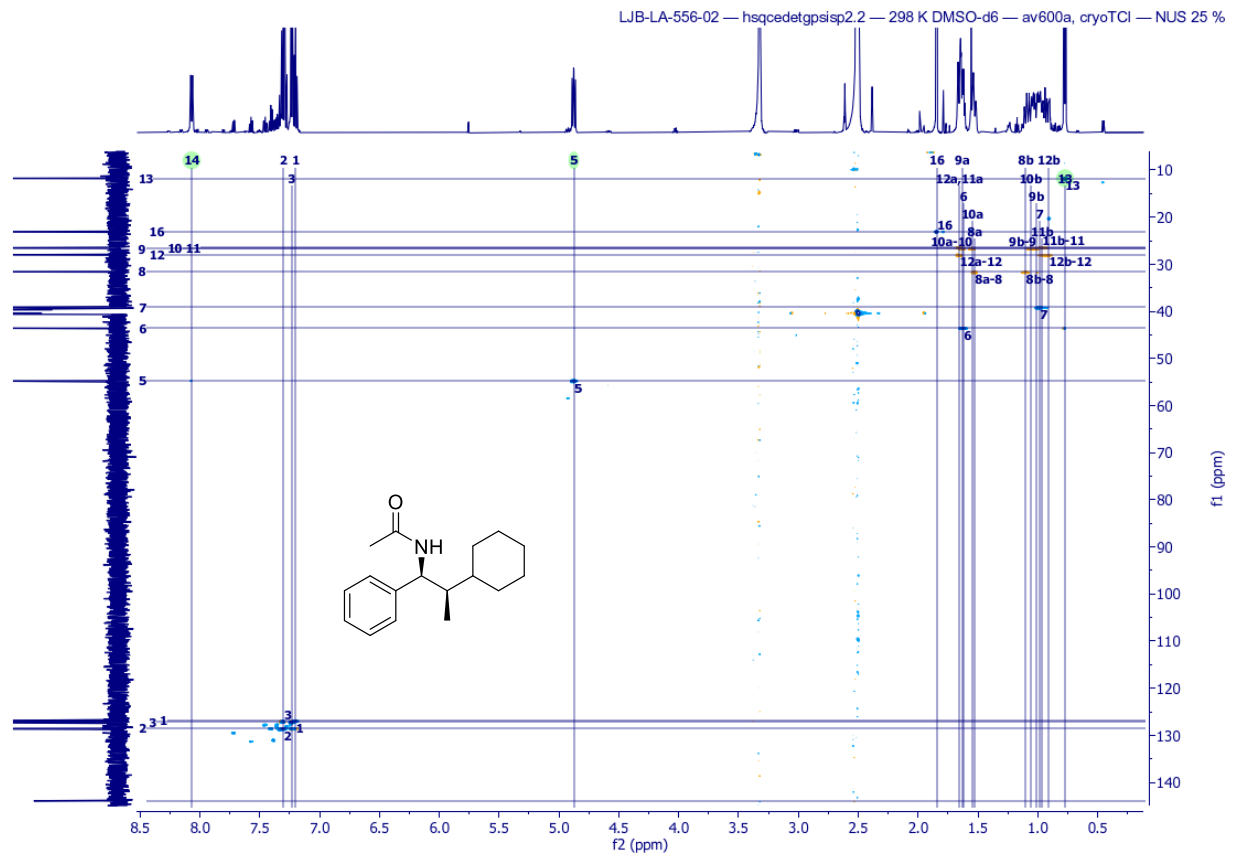
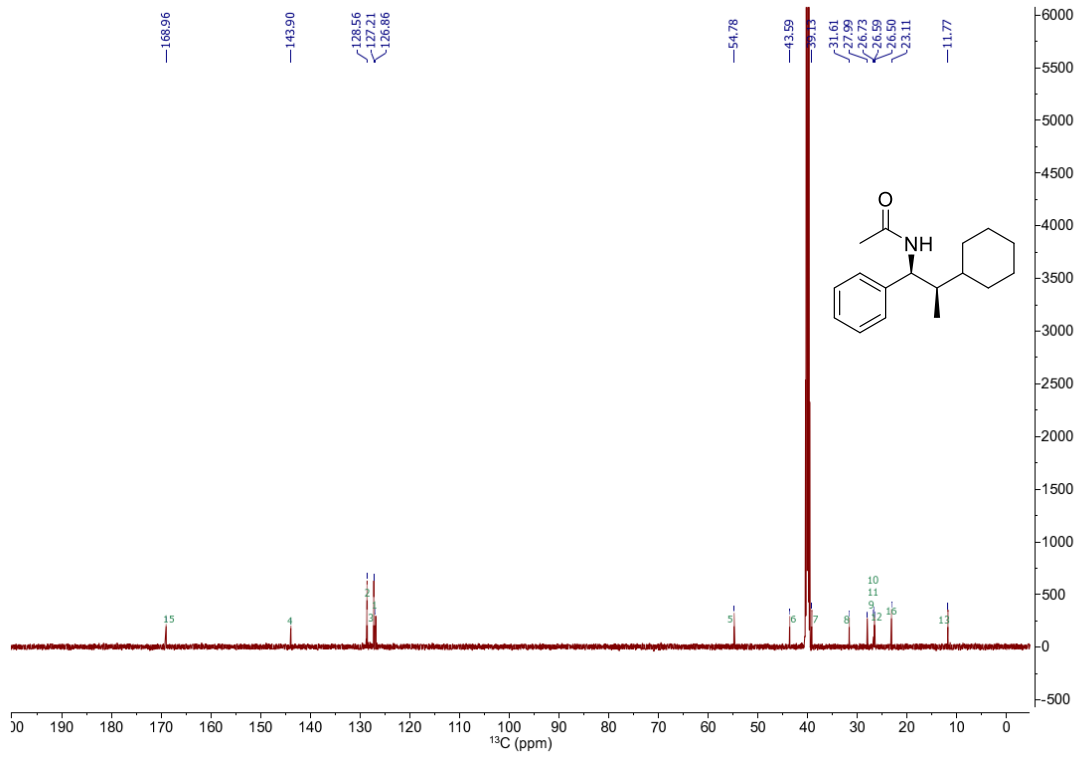
C and H 9, 10 and 11 could not be assigned unequivocally and are possibly to be interchanged.

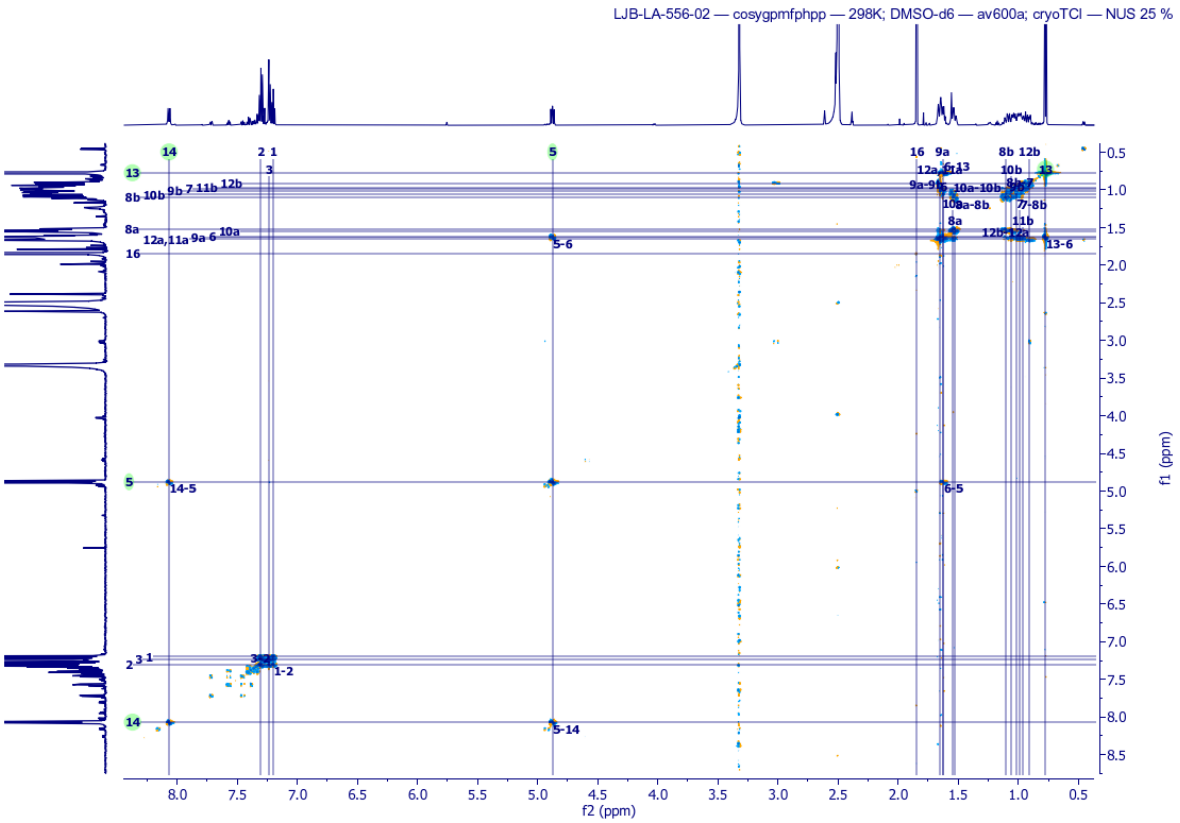
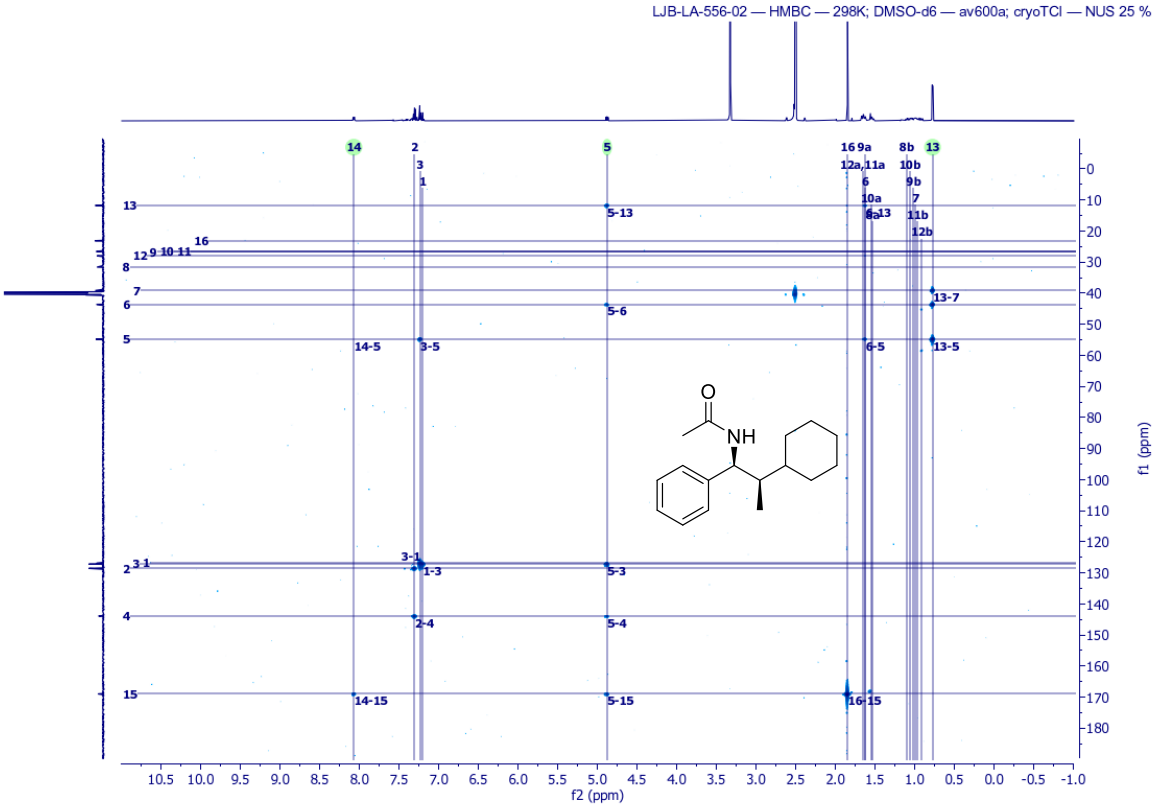
See for comparison: LJB-LA-556-01

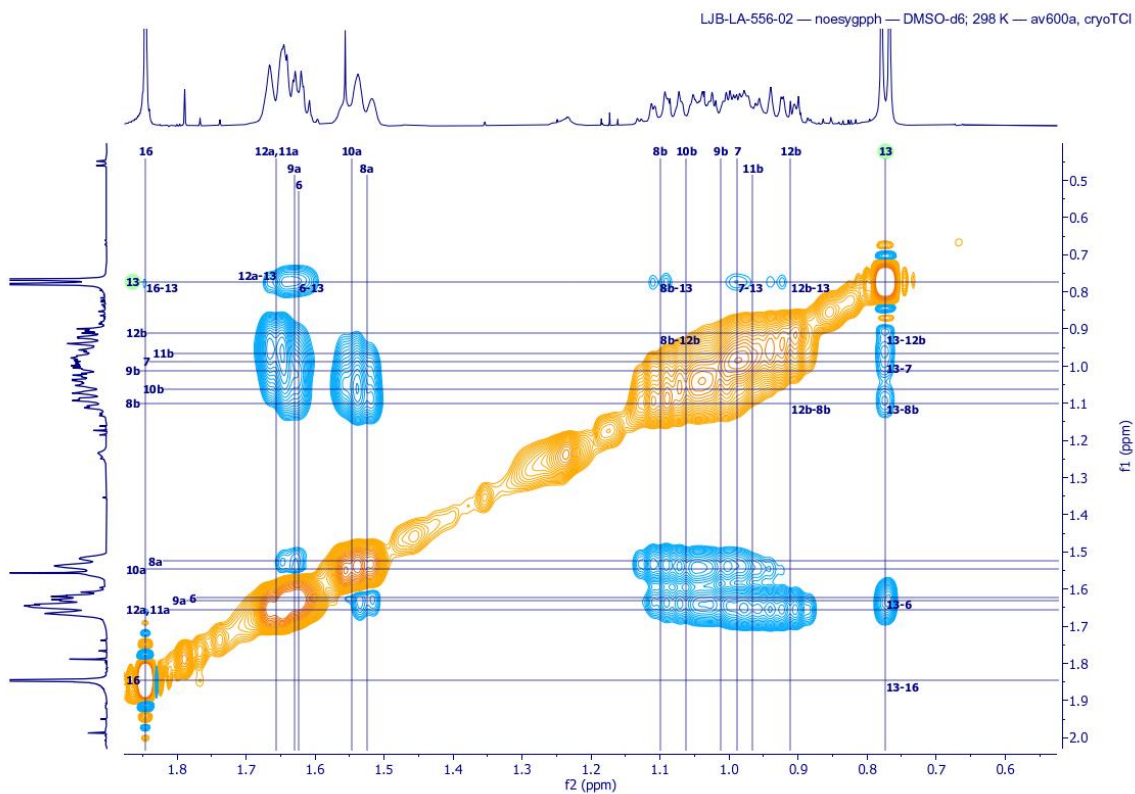
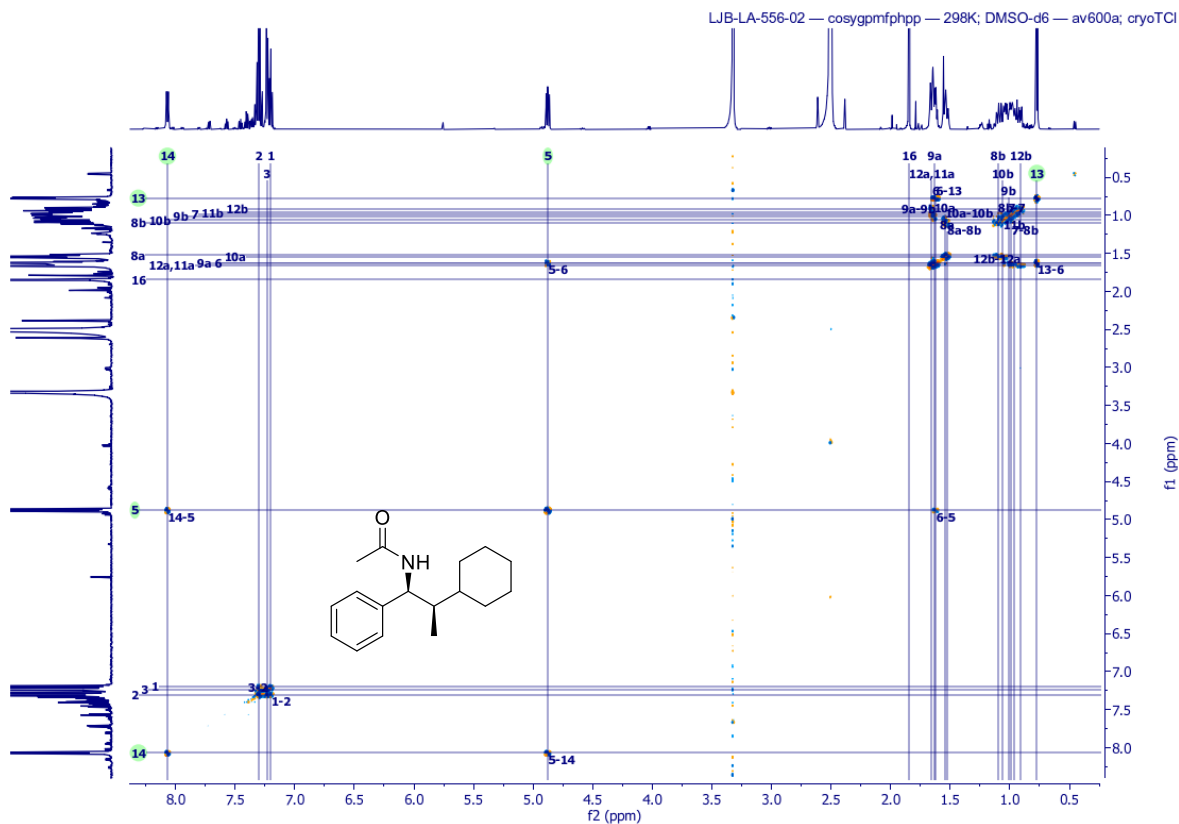
| Atom | δ (ppm) | J | HSQC | HMQC | COSY | NOESY | ¹⁵ N-HMBC |
|------|----------------|--------------------------------|----------|-----------------|-------|----------------------------------|----------------------|
| C1 | 126.86 | | 1 | 3 | | | |
| H1 | 7.200 | m | 1 | 3 | 2 | | |
| C2 | 128.56 | | 2 | | | | |
| H2 | 7.304 | m | 2 | 4 | 1, 3 | | |
| C3 | 127.21 | | 3 | 1, 5 | | | |
| H3 | 7.232 | m | 3 | 1, 5 | 2 | 5, 6, 7, 12a, 13, 14, 16 | |
| C4 | 143.90 | | | 2, 5 | | | |
| C5 | 54.78 | | 5 | 3, 6, 13, 14 | | | |
| H5 | 4.877 | d 9.30(14), d 7.60(6) | 5 | 3, 4, 6, 13, 15 | 6, 14 | 3, 6, 7, 8a, 12a, 13, 14 | |
| C6 | 43.59 | | 6 | 5, 13 | | | |
| H6 | 1.624 | d 7.60(5), q 6.90(13), d ~5(7) | 6 | 5, 13 | 5, 13 | 3, 5, 13, 14 | |
| C7 | 39.13 | | 7 | 13 | | | |
| H7 | 0.988 | m (ax) | 7 | | 8b | 3, 5, 13, 14 | |
| C8 | 31.61 | | 8a, 8b | | | | |
| H8a | 1.525 | m (eq) | 8 | | 8b | 5 | |
| H8b | 1.100 | m (ax) | 8 | | 7, 8a | 12b, 13 | |
| C9 | 26.73 | | 9a, 9b | | | | |
| H9a | 1.630 | m (eq) | 9 | | 9b | | |
| H9b | 1.012 | m (ax) | 9 | | 9a | | |
| C10 | 26.59 | | 10a, 10b | | | | |
| H10a | 1.547 | m (eq) | 10 | | 10b | | |
| H10b | 1.062 | m (ax) | 10 | | 10a | | |
| C11 | 26.50 | | 11a, 11b | | | | |
| H11a | 1.656 | m (eq) | 11 | | 11b | | |
| H11b | 0.966 | m (ax) | 11 | | 11a | | |
| C12 | 27.99 | | 12a, 12b | | | | |
| H12a | 1.656 | m (eq) | 12 | | 12b | 3, 5, 13 | |
| H12b | 0.911 | m (ax) | 12 | | 12a | 8b, 13 | |
| C13 | 11.77 | | 13 | 5, 6 | | | |
| H13 | 0.773 | d 6.90(6) | 13 | 5, 6, 7 | 6 | 3, 5, 6, 7, 8b, 12a, 12b, 14, 16 | |
| H14 | -254.3 | | | | | | 14 |
| H14 | 8.068 | d 9.30(5) | | 5, 15 | 5 | 3, 5, 6, 7, 13, 16 | 14 |
| C15 | 168.96 | | | 5, 14, 16 | | | |
| C16 | 23.11 | | 16 | | | | |
| H16 | 1.846 | s | 16 | 15 | | 3, 13, 14 | |

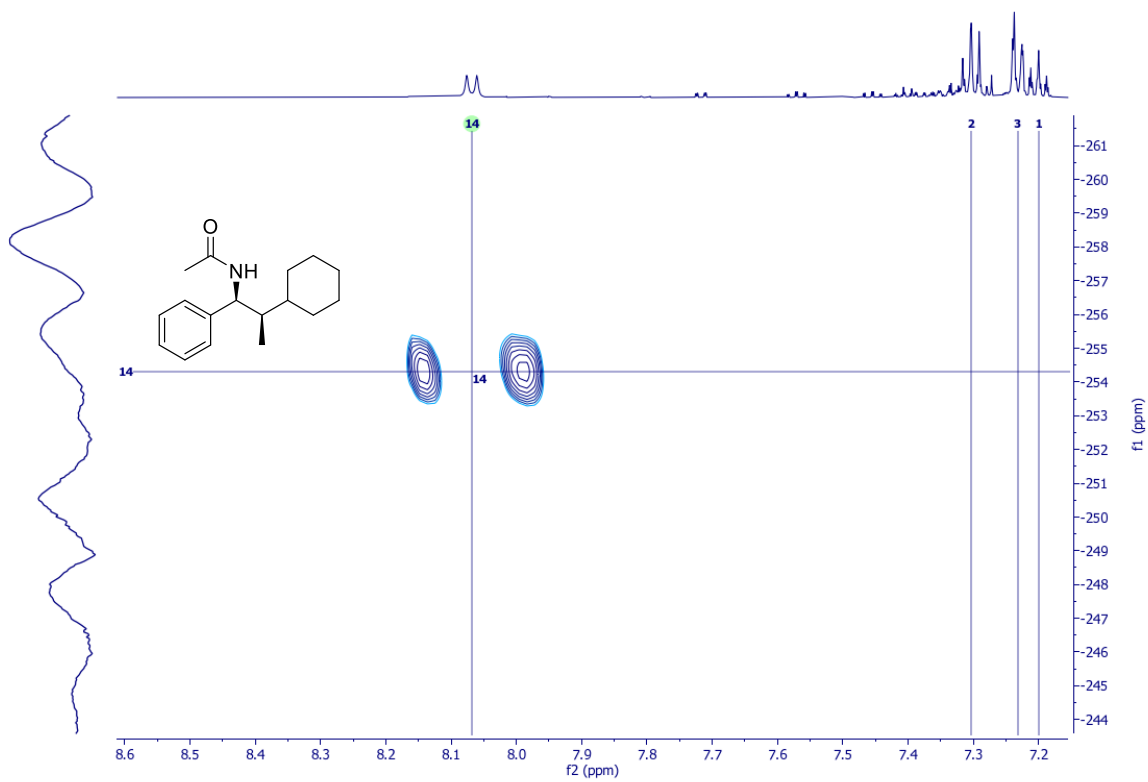
LJB-LA-556-02 1H NMR





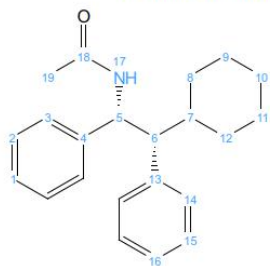




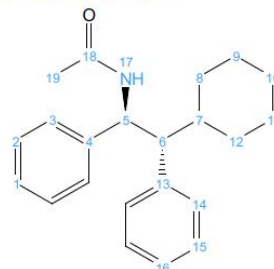


LJB-LA-557
DMSO-d₆; 298 K; 2 mg; AV600a

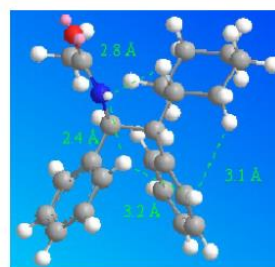
The NMR-data support the following relative stereochemistry:



Major Component (*rel*-R,R)



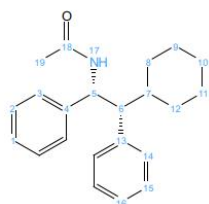
Minor Component (*rel*-S,R)



(2:1)

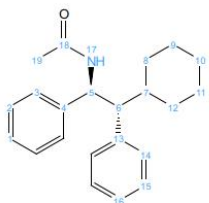
Click on the pictures to view the 3D-models in Chem3D.

Warning: the depicted atom distances have been generated by Chem3D and are not based on experimental values! They are merely a means to qualitatively illustrate the spatial arrangement derived from the NOESY correlations.



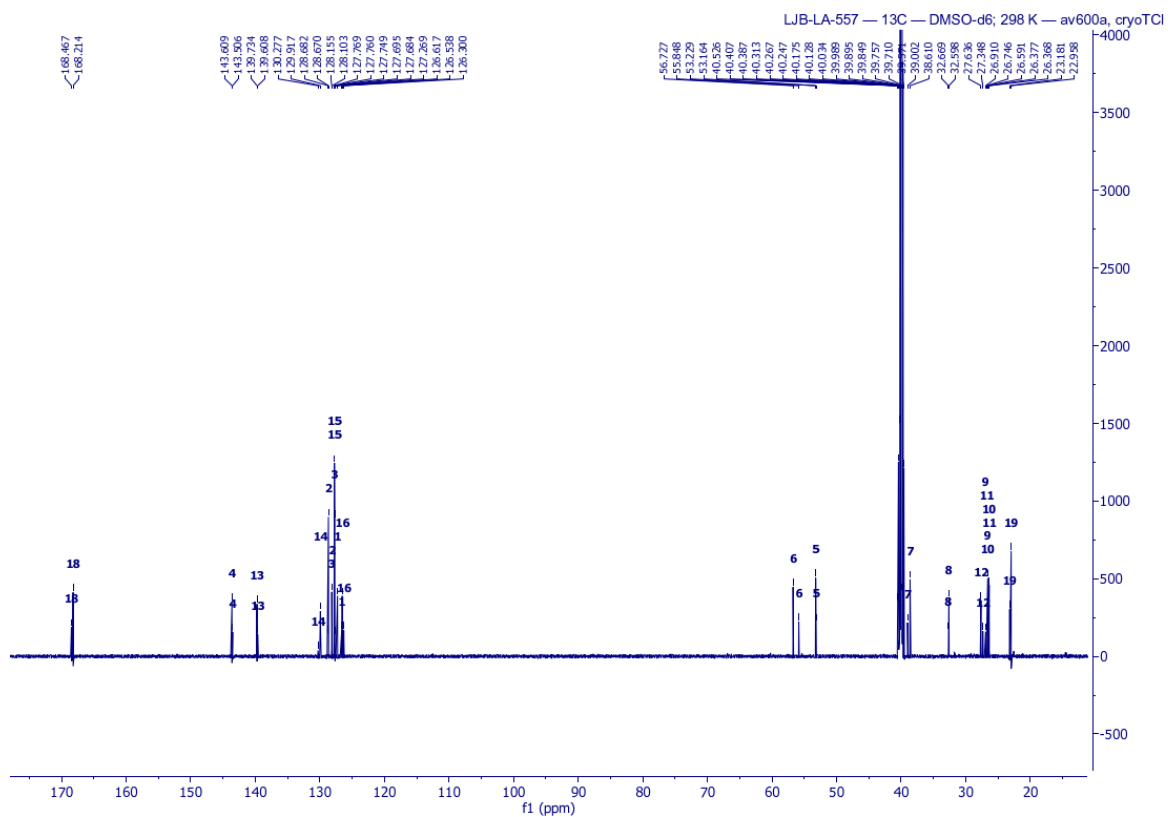
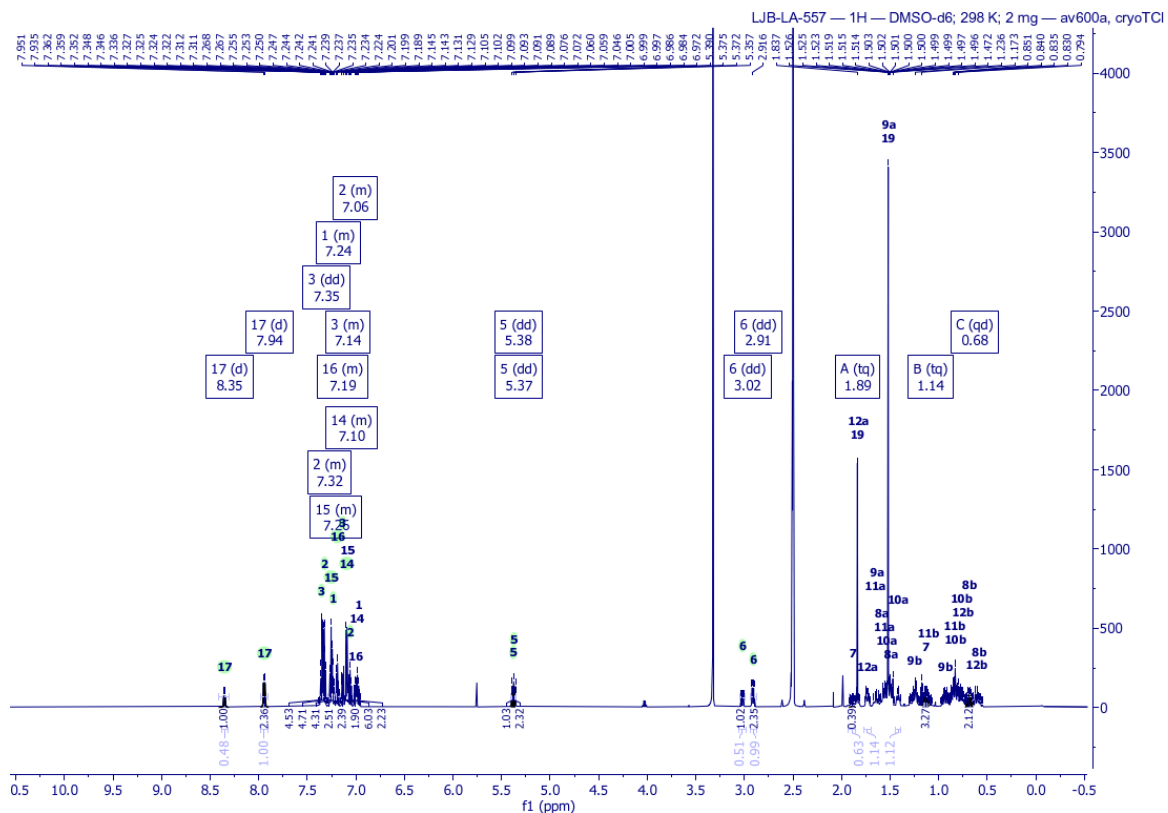
Major Component (*rel*-R,R)

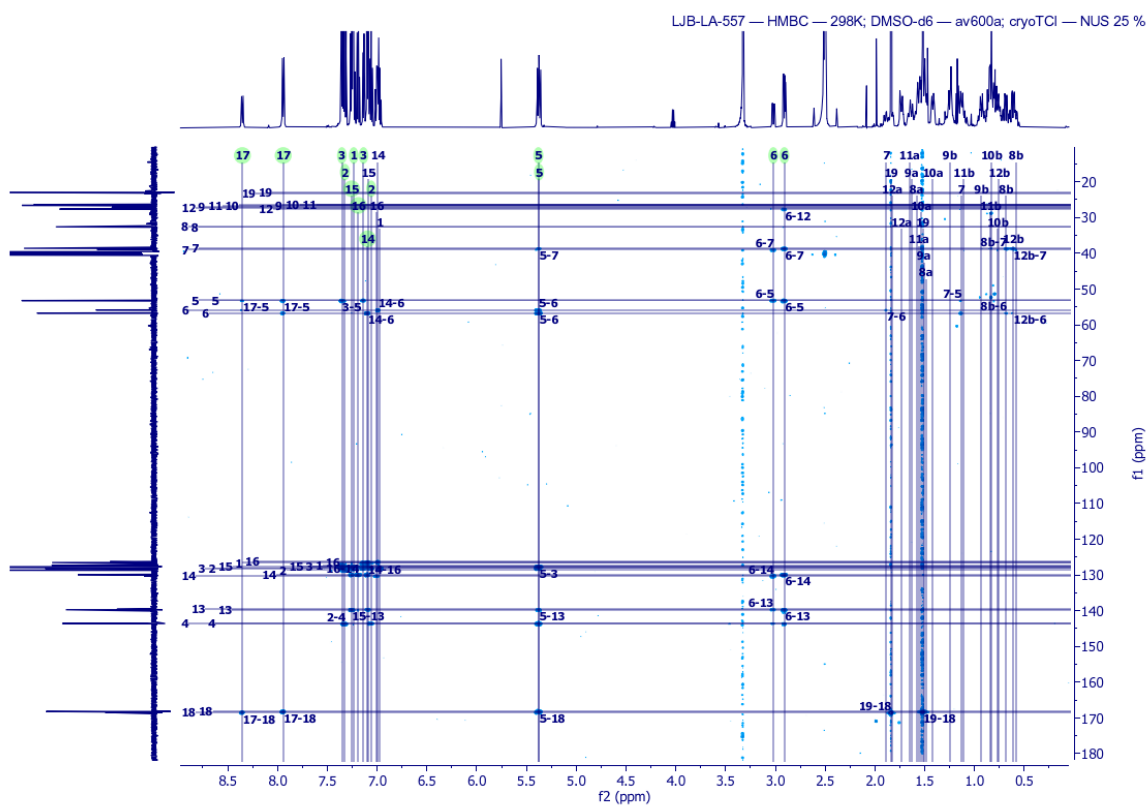
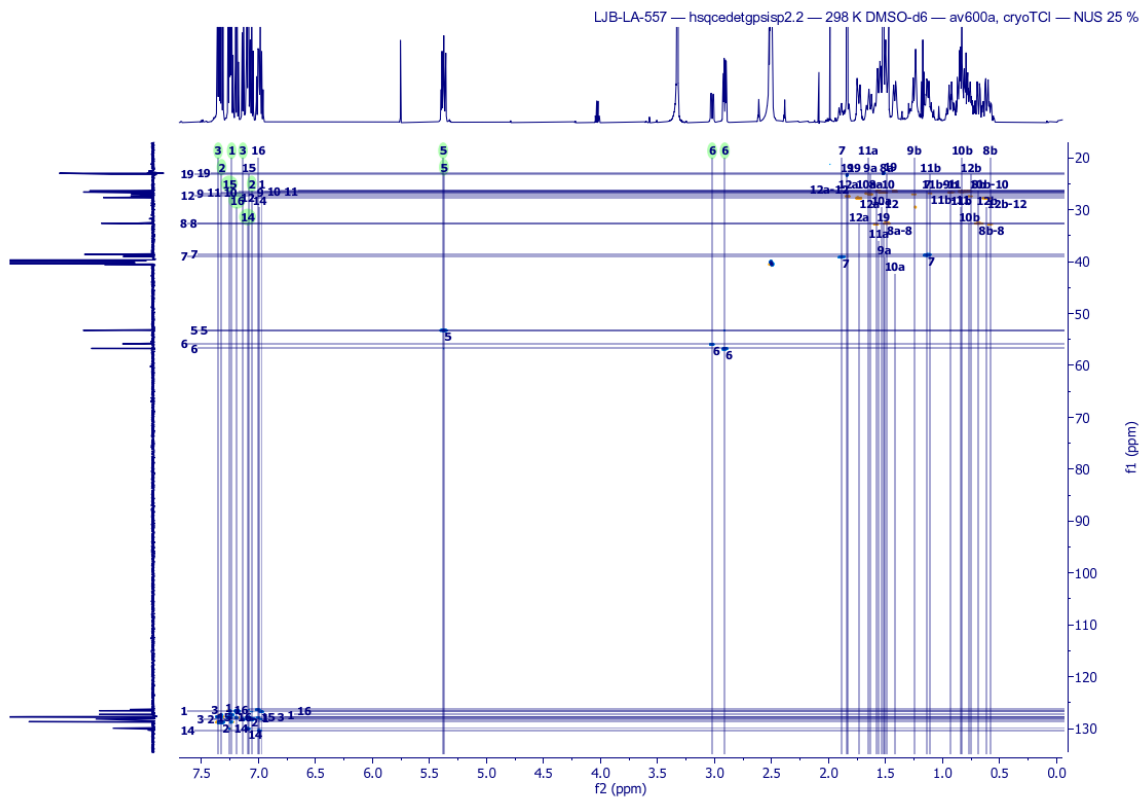
| Atom | δ (ppm) | J | NOESY | COSY | HSQC | HMQC | ^{15}N -HMBC |
|------|----------------|----------------------|-----------------------------------|----------------------------|----------|------------------------|-----------------------|
| C1 | 127.27 | | | | 1 | 3 | |
| H1 | 7.235 | m | | 2, 3 | 1 | 3 | |
| C2 | 128.67 | | | | 2 | | |
| H2 | 7.324 | m | | 1, 3 | 2 | 4 | |
| C3 | 127.68 | | | | 3 | 1, 5 | |
| H3 | 7.354 | m | 5, 6, 7, 12a, 17, 19 | 1, 2 | 3 | 1, 5 | |
| C4 | 143.61 | | | | | 2, 5, 6 | |
| C5 | 53.23 | | | | 5 | 3, 6, 7, 17 | |
| H5 | 5.374 | d 10.9(6), d 9.3(17) | 3, 6, 7, 8a, 12a, 14, 17 | 6, 17 | 5 | 3, 4, 6, 7, 13, 18 | |
| C6 | 56.73 | | | | 6 | 5, 7, 8b, 12b, 14, 17 | |
| H6 | 2.910 | d 10.9(5), d 4.3(7) | 3, 5, 7, 8a, 14, 17 | 5, 7 | 6 | 4, 5, 7, 12, 13, 14 | |
| C7 | 38.61 | | | | 7 | 5, 6, 8b, 9b, 11b, 12b | |
| H7 | 1.136 | m (ax) | 3, 5, 6, 8a, 9b, 11b, 12a, 14 | 6, 8a, 8b, 12a, 12b | 7 | 5, 6 | |
| C8 | 32.67 | | | | 8a, 8b | | |
| H8a | 1.489 | m (eq) | 5, 6, 7, 8b, 14 | 7, 8b, 9a, 9b, 12a | 8 | | |
| H8b | 0.683 | m (ax) | 8a, 12b, 14 | 7, 8a, 9a, 9b | 8 | 6, 7 | |
| C9 | 26.59 | | | | 9a, 9b | | |
| H9a | 1.510 | m (eq) | 9b, 10b | 8a, 8b, 9b, 10a, 10b | 9 | | |
| H9b | 0.931 | m (ax) | 7, 9a, 10a | 8a, 8b, 9a, 10a, 10b | 9 | 7 | |
| C10 | 26.38 | | | | 10a, 10b | | |
| H10a | 1.419 | m (eq) | 9b, 10b, 11a, 11b | 9a, 9b, 10b, 11a, 11b, 12a | 10 | | |
| H10b | 0.767 | m (ax) | 9a, 10a, 11a, 12b | 9a, 9b, 10a, 11a, 11b | 10 | | |
| C11 | 26.37 | | | | 11a, 11b | | |
| H11a | 1.561 | m (eq) | 10a, 10b, 11b, 12a | 10a, 10b, 11b, 12a, 12b | 11 | | |
| H11b | 0.840 | m (ax) | 7, 10a, 11a, 12a | 10a, 10b, 11a, 12a, 12b | 11 | 7 | |
| C12 | 27.64 | | | | 12a, 12b | 6 | |
| H12a | 1.735 | m (eq) | 3, 5, 7, 11a, 11b, 12b, 14 | 7, 8a, 10a, 11a, 11b, 12b | 12 | | |
| H12b | 0.613 | m (ax) | 8b, 10b, 12a, 14 | 7, 11a, 11b, 12a | 12 | 6, 7 | |
| C13 | 139.73 | | | | | 5, 6, 15 | |
| C14 | 129.92 | | | | 14 | 6, 15, 16 | |
| H14 | 7.096 | m | 5, 6, 7, 8a, 8b, 12a, 12b, 17, 19 | 15, 16 | 14 | 6, 16 | |
| C15 | 127.75 | | | | 15 | | |
| H15 | 7.256 | m | 19 | 14, 16 | 15 | 13, 14 | |
| C16 | 126.54 | | | | 16 | 14 | |
| H16 | 7.189 | m | | 14, 15 | 16 | 14 | |
| N17 | -248.8 | | | | | | 17 |
| H17 | 7.943 | d 9.3(5) | 3, 5, 6, 14, 19 | 5 | | 5, 6, 18 | 17 |
| C18 | 168.21 | | | | | 5, 17, 19 | |
| C19 | 22.96 | | | | 19 | | |
| H19 | 1.519 | s | 3, 14, 15, 17 | | 19 | 18 | |

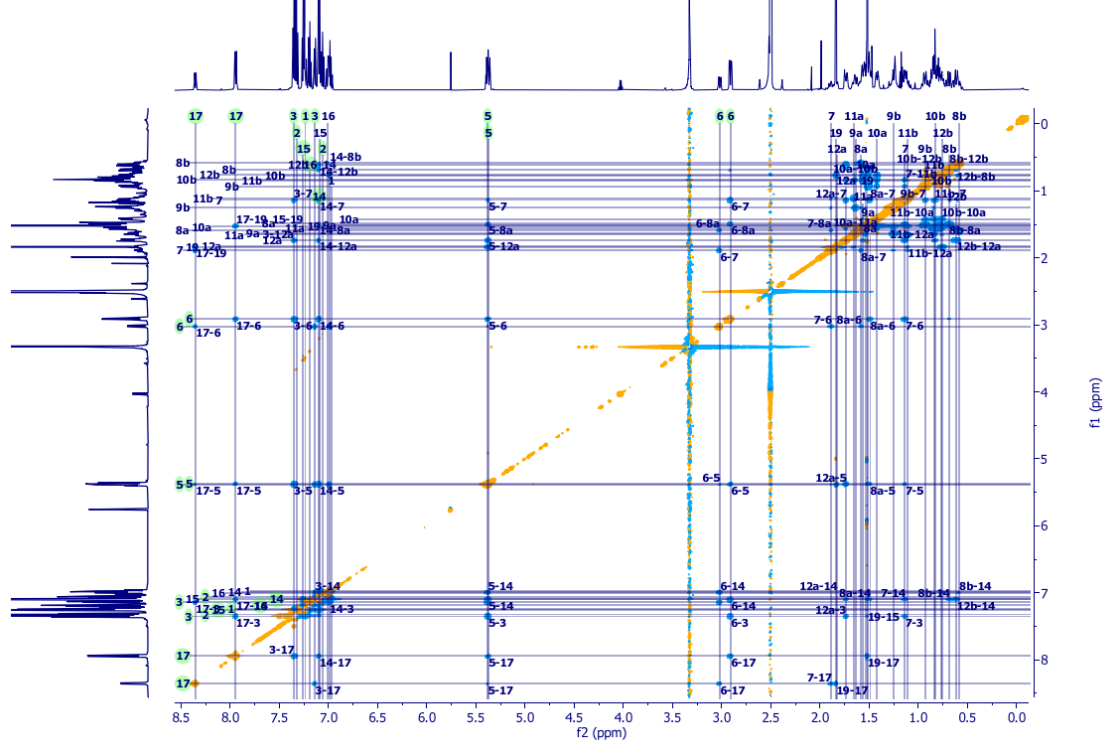
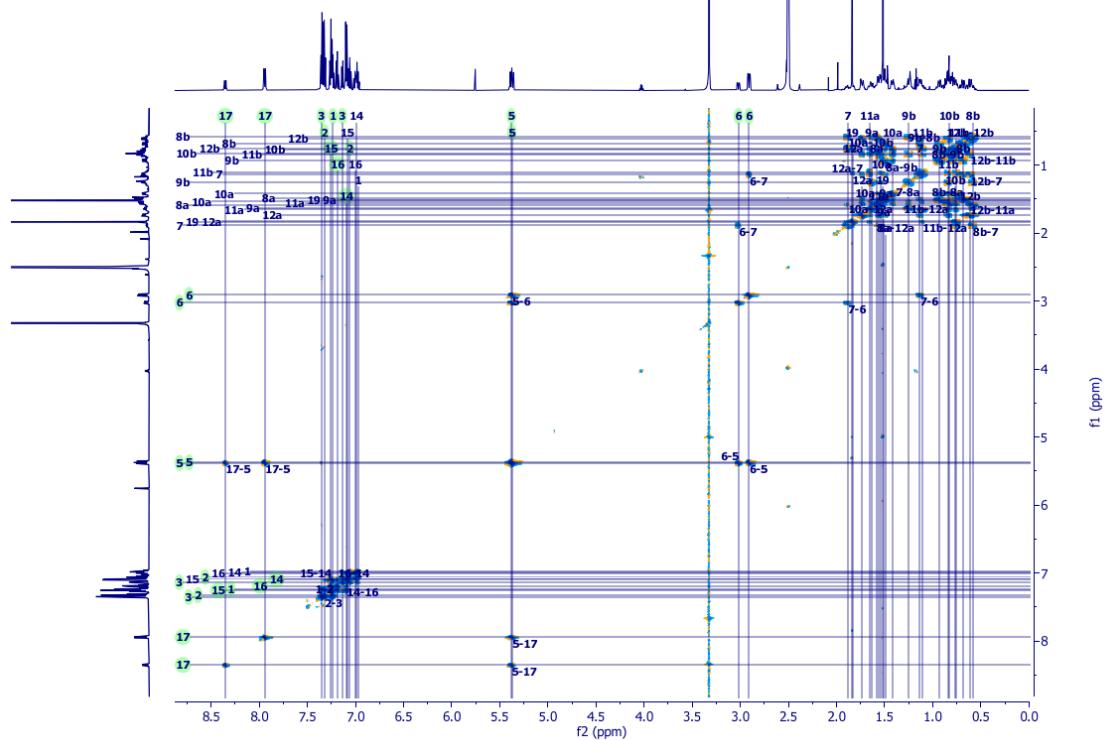


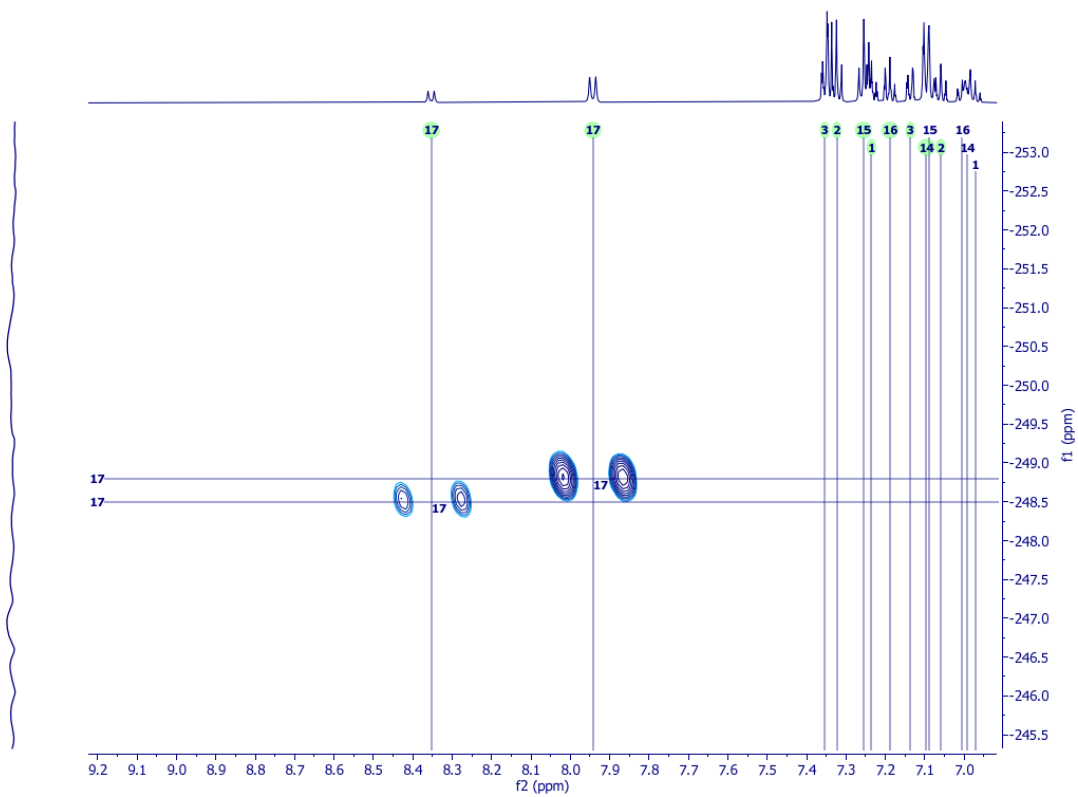
Minor Component (*rel*-S,R)

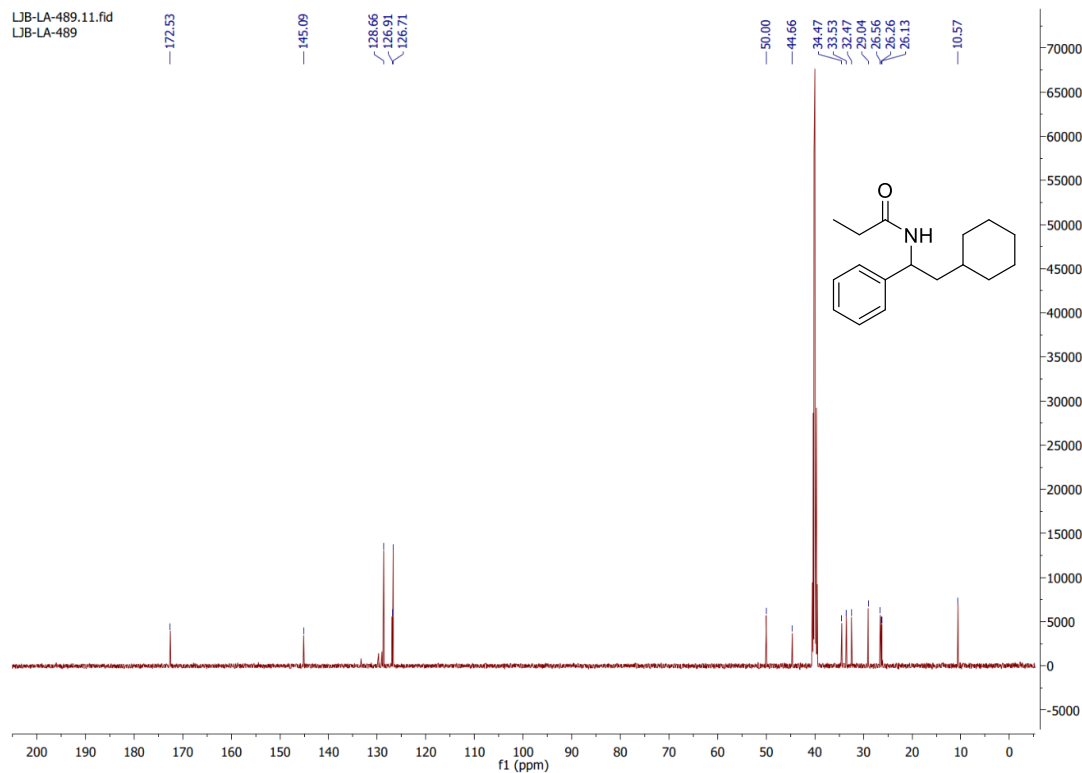
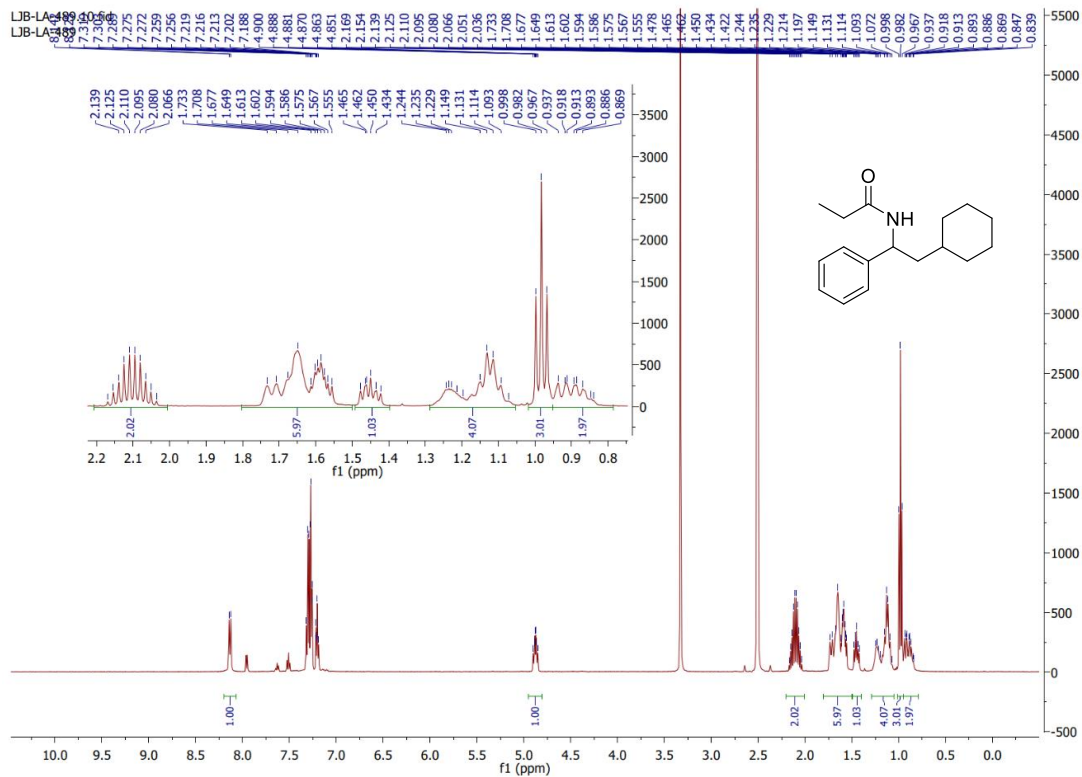
| Atom | δ (ppm) | J | NOESY | COSY | HSQC | HMQC | ^{15}N -HMBC |
|------|----------------|------------------------|-----------------------|-------------------------|----------|--------------------|-----------------------|
| C1 | 126.62 | | | | 1 | 3 | |
| H1 | 6.972 | m | | 2, 3 | 1 | 3 | |
| C2 | 128.10 | | | | 2 | | |
| H2 | 7.059 | m | | 1, 3 | 2 | 4 | |
| C3 | 128.16 | | | | 3 | 1, 5 | |
| H3 | 7.137 | m | 5, 6, 14, 17 | 1, 2 | 3 | 1, 5 | |
| C4 | 143.51 | | | | | 2, 5, 6 | |
| C5 | 53.16 | | | | 5 | 3, 6, 17 | |
| H5 | 5.380 | d 9.30(17), d 11.70(6) | 3, 6, 7, 12a, 14, 17 | 6, 17 | 5 | 3, 4, 6, 7, 13, 18 | |
| C6 | 55.85 | | | | 6 | 5, 7, 14, 17 | |
| H6 | 3.021 | d 11.70(5), d 3.90(7) | 3, 5, 7, 8a, 14, 17 | 5, 7 | 6 | 4, 5, 7, 13, 14 | |
| C7 | 39.00 | | | | 7 | 5, 6, 8b, 12b | |
| H7 | 1.886 | m (ax) | 5, 6, 8a, 9b, 11b, 17 | 6, 8a, 8b, 12a, 12b | 7 | 6 | |
| C8 | 32.60 | | | | 8a, 8b | | |
| H8a | 1.583 | m (eq) | 6, 7, 8b | 7, 8b, 9a, 9b, 12a | 8 | | |
| H8b | 0.583 | m (ax) | 8a, 12b, 14 | 7, 8a, 9a, 9b | 8 | 7 | |
| C9 | 26.91 | | | | 9a, 9b | | |
| H9a | 1.632 | m (eq) | 9b, 10b | 8a, 8b, 9b, 10a, 10b | 9 | | |
| H9b | 1.249 | m (ax) | 7, 9a | 8a, 8b, 9a, 10a, 10b | 9 | | |
| C10 | 26.59 | | | | 10a, 10b | | |
| H10a | 1.536 | m (eq) | 10b | 9a, 9b, 10b, 11a, 11b | 10 | | |
| H10b | 0.830 | m (ax) | 9a, 10a, 11a | 9a, 9b, 10a, 11a, 11b | 10 | | |
| C11 | 26.75 | | | | 11a, 11b | | |
| H11a | 1.655 | m (eq) | 10b, 11b | 10a, 10b, 11b, 12a, 12b | 11 | | |
| H11b | 1.109 | m (ax) | 7, 11a, 12a | 10a, 10b, 11a, 12a, 12b | 11 | | |
| C12 | 27.35 | | | | 12a, 12b | | |
| H12a | 1.828 | m (eq) | 5, 11b, 12b, 14 | 7, 8a, 11a, 11b, 12b | 12 | | |
| H12b | 0.754 | m (ax) | 8b, 12a, 14 | 7, 11a, 11b, 12a | 12 | 7 | |
| C13 | 139.61 | | | | | 5, 6, 15 | |
| C14 | 130.28 | | | | 14 | 6, 16 | |
| H14 | 6.992 | m | 3, 5, 6, 8b, 12a, 12b | 15 | 14 | 6, 16 | |
| C15 | 127.75 | | | | 15 | | |
| H15 | 7.088 | m | | 14, 16 | 15 | 13 | |
| C16 | 126.30 | | | | 16 | 14 | |
| H16 | 7.005 | m | | 15 | 16 | 14 | |
| N17 | -248.5 | | | | | | 17 |
| H17 | 8.353 | d 9.30(5) | 3, 5, 6, 7, 19 | 5 | | 5, 6, 18 | 17 |
| C18 | 168.47 | | | | | 5, 17, 19 | |
| C19 | 23.18 | | | | 19 | | |
| H19 | 1.837 | s | 17 | | 19 | 18 | |



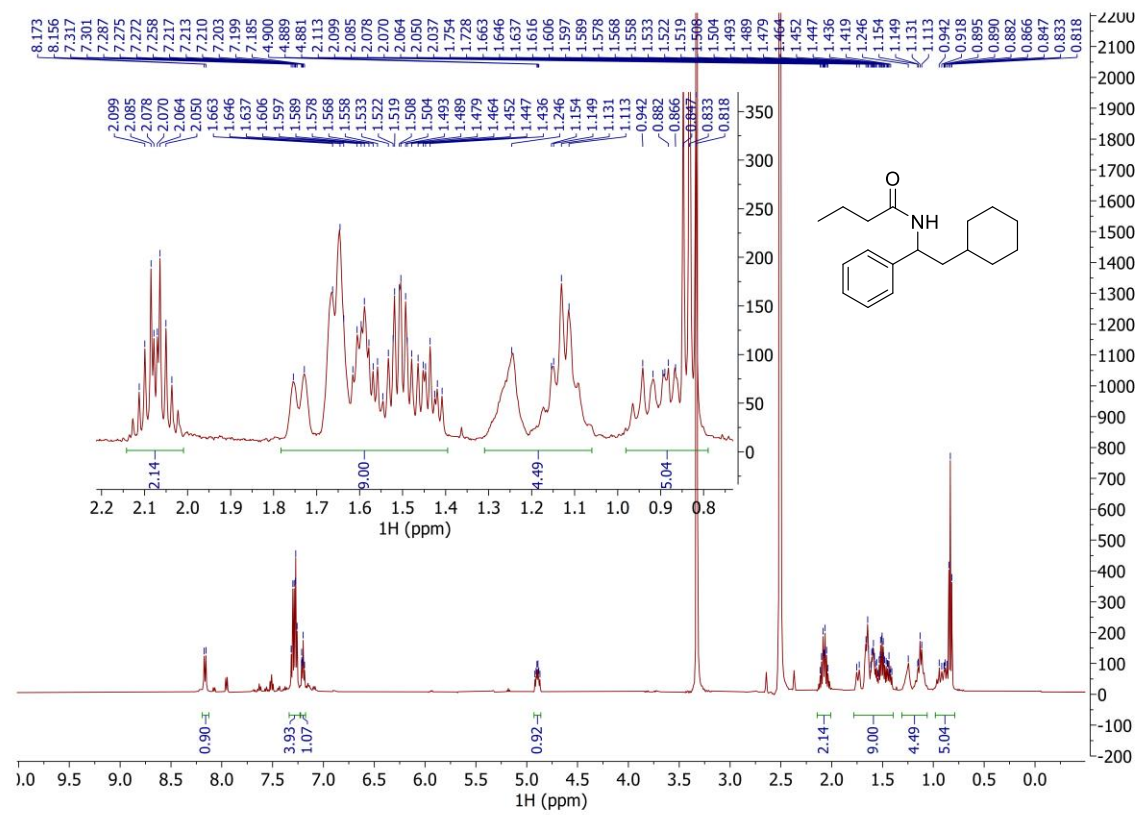
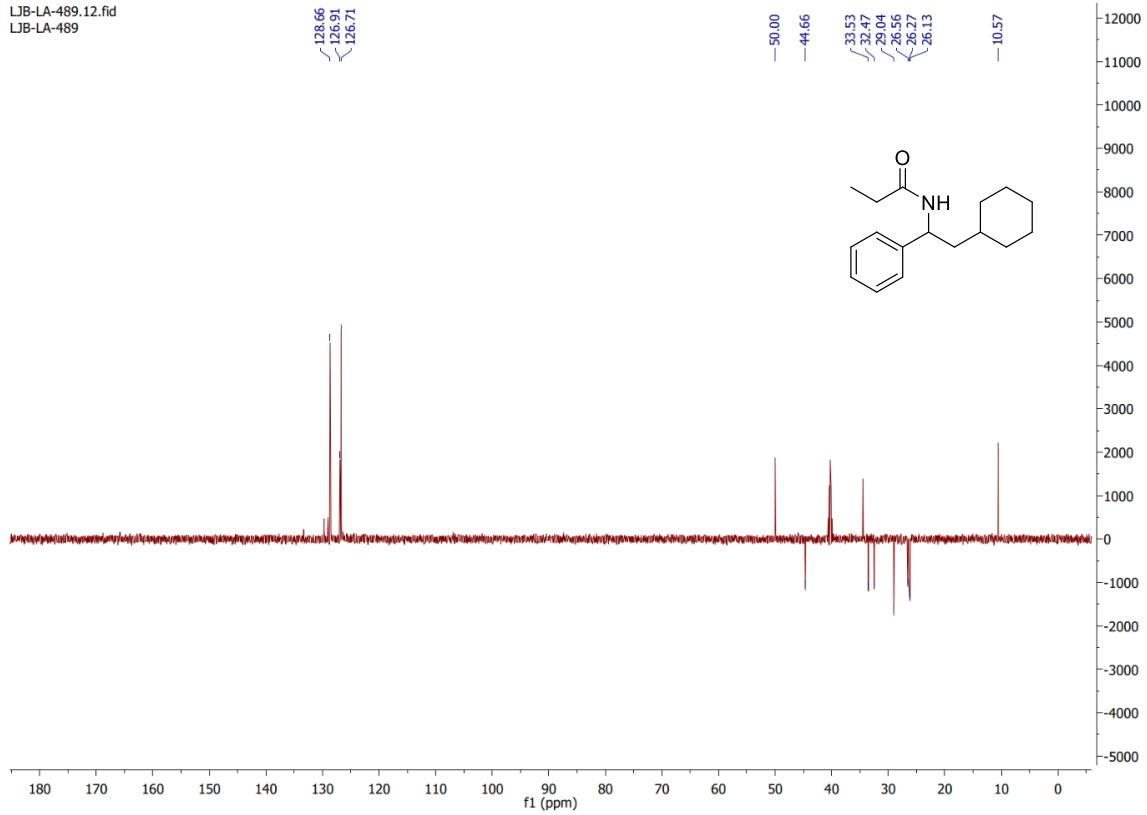


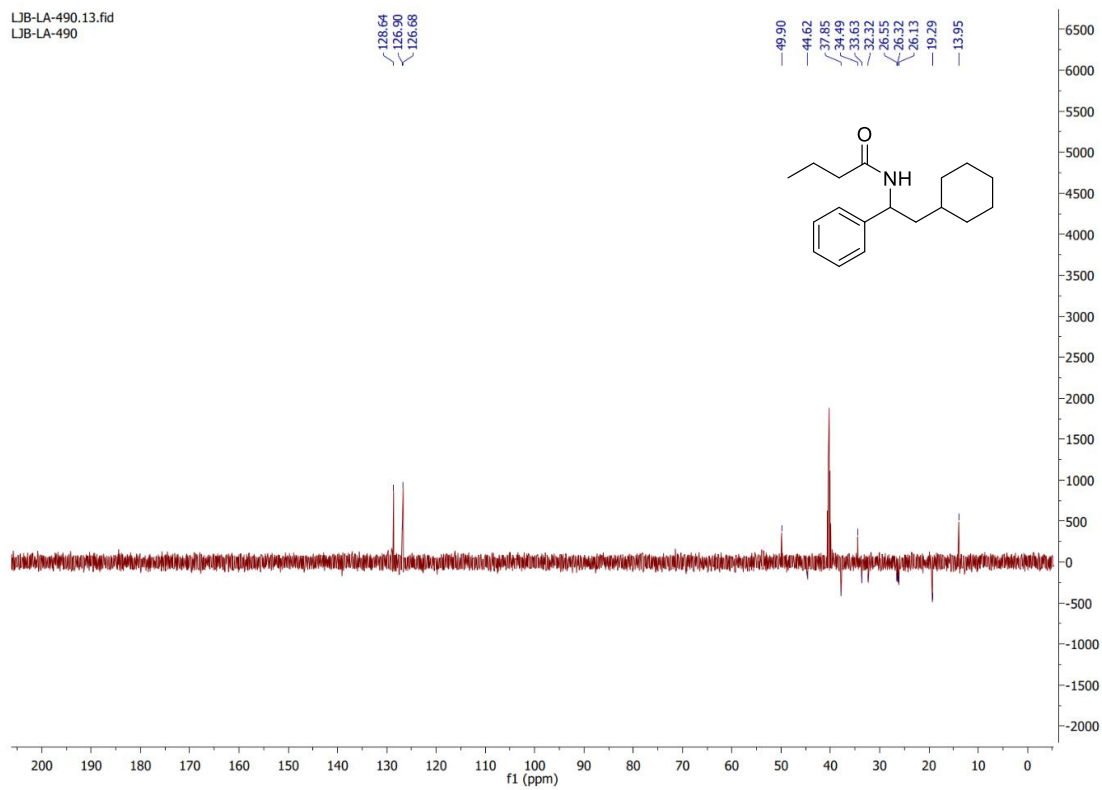
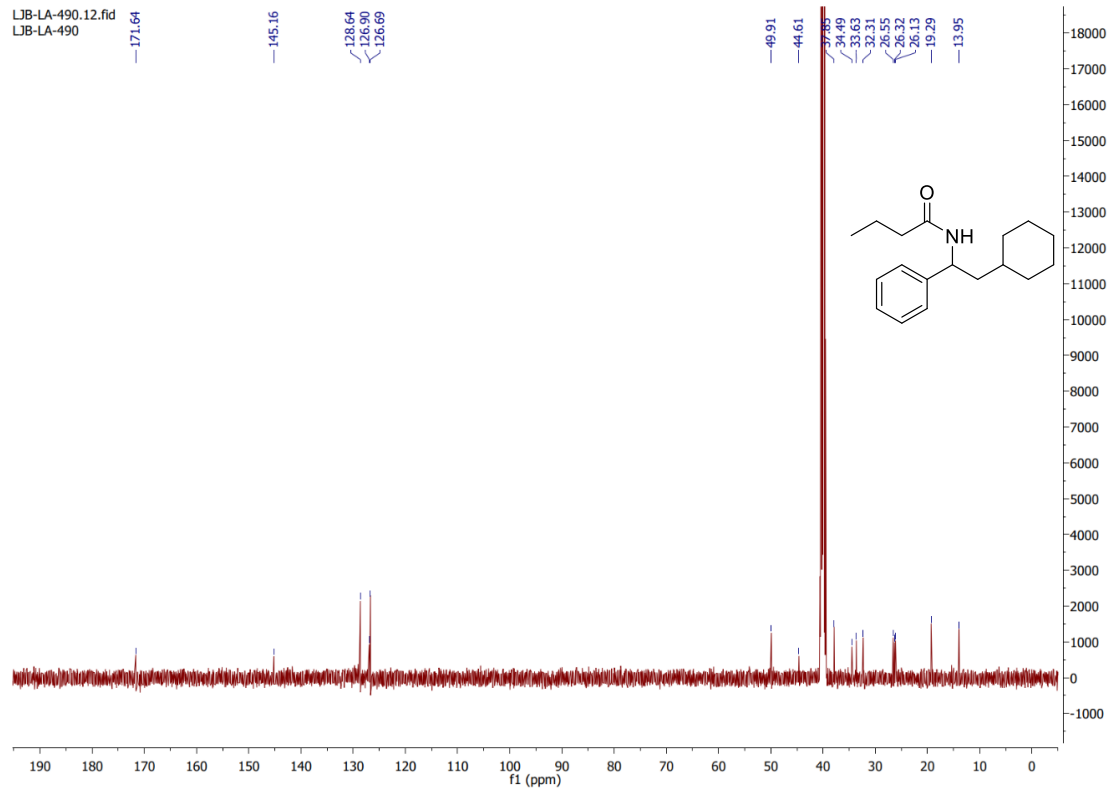


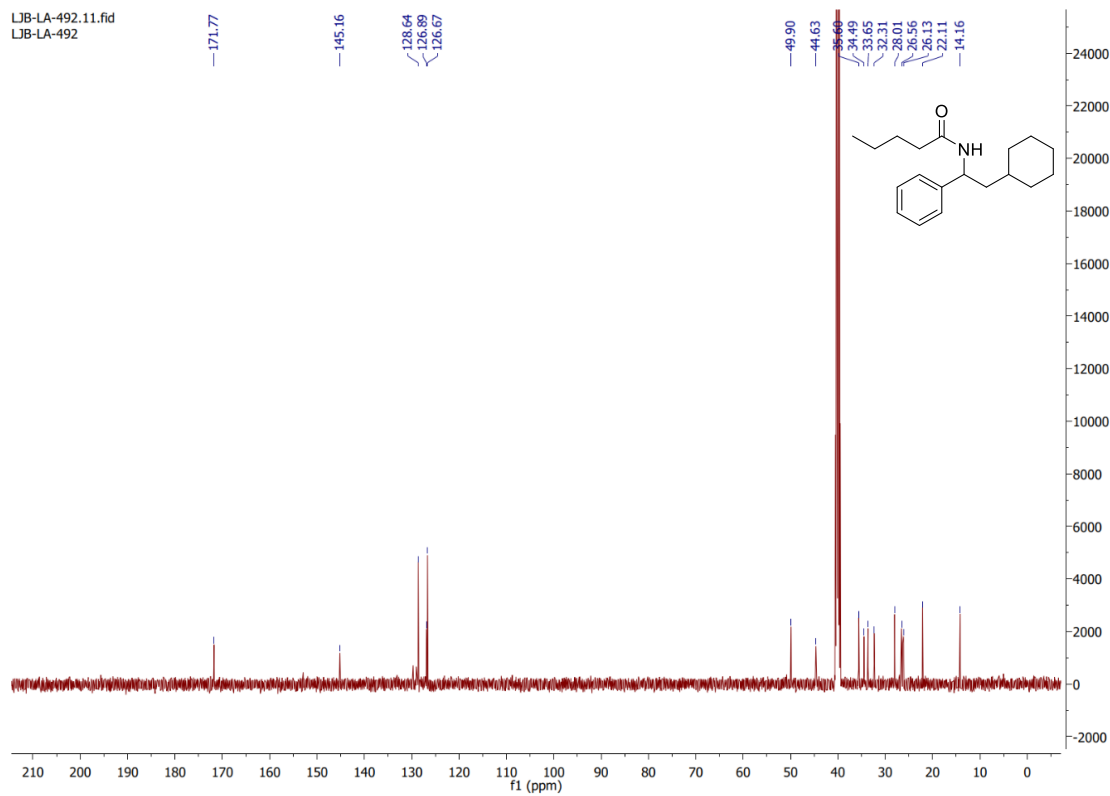
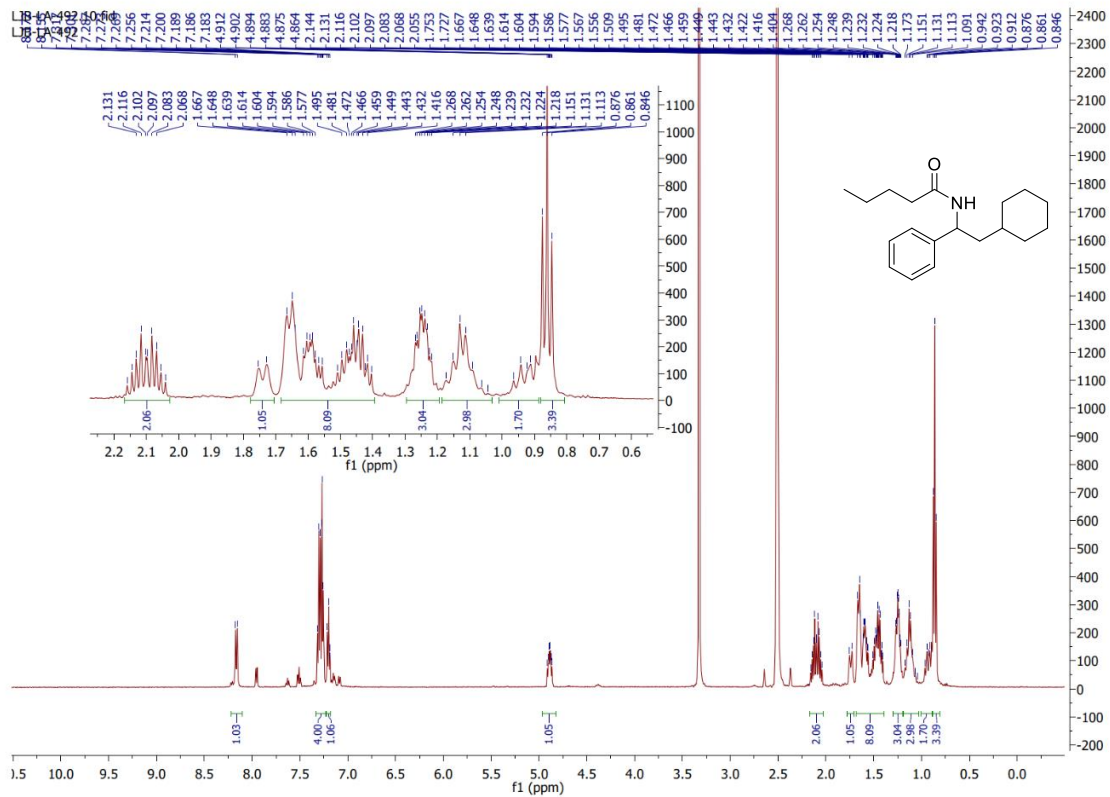




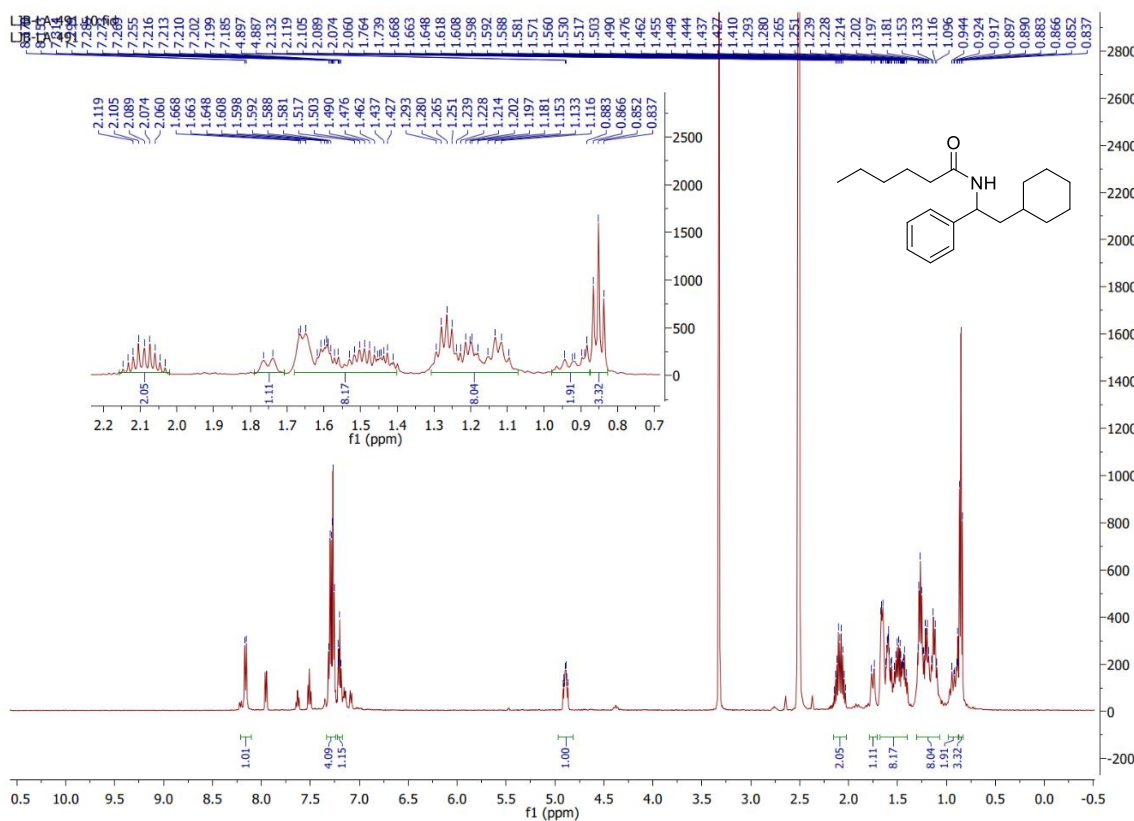
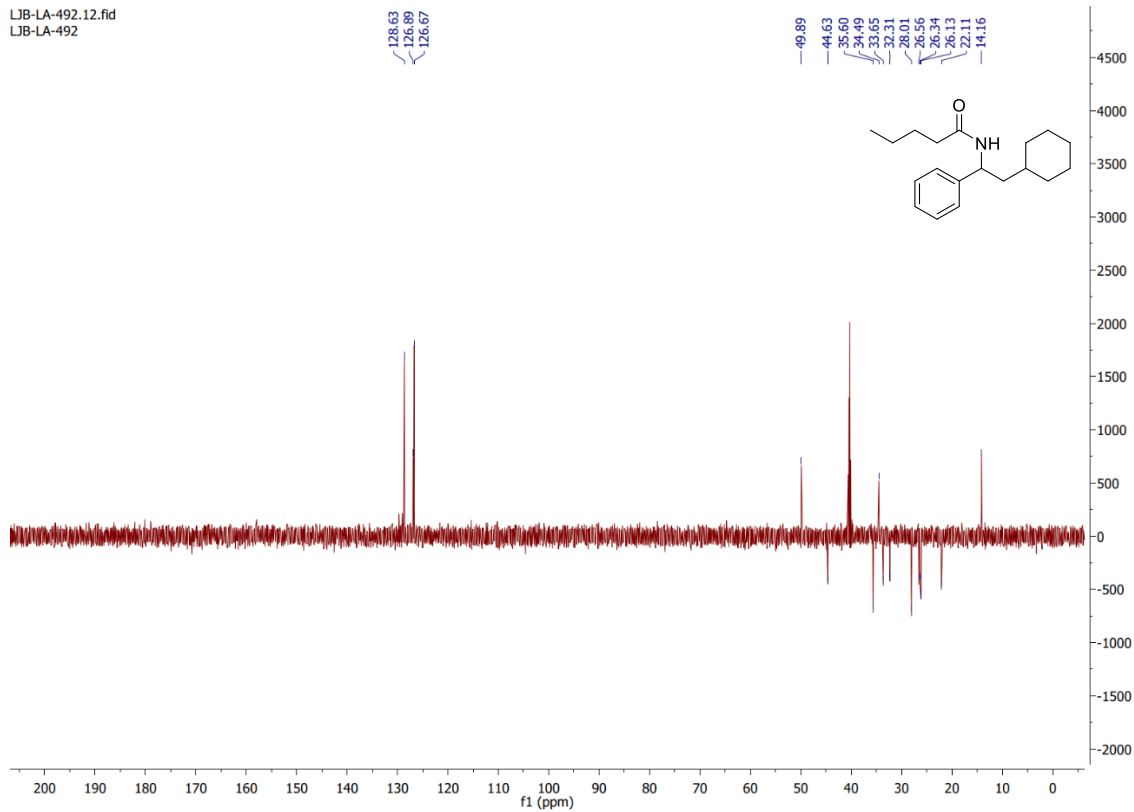
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LJB-LA-489



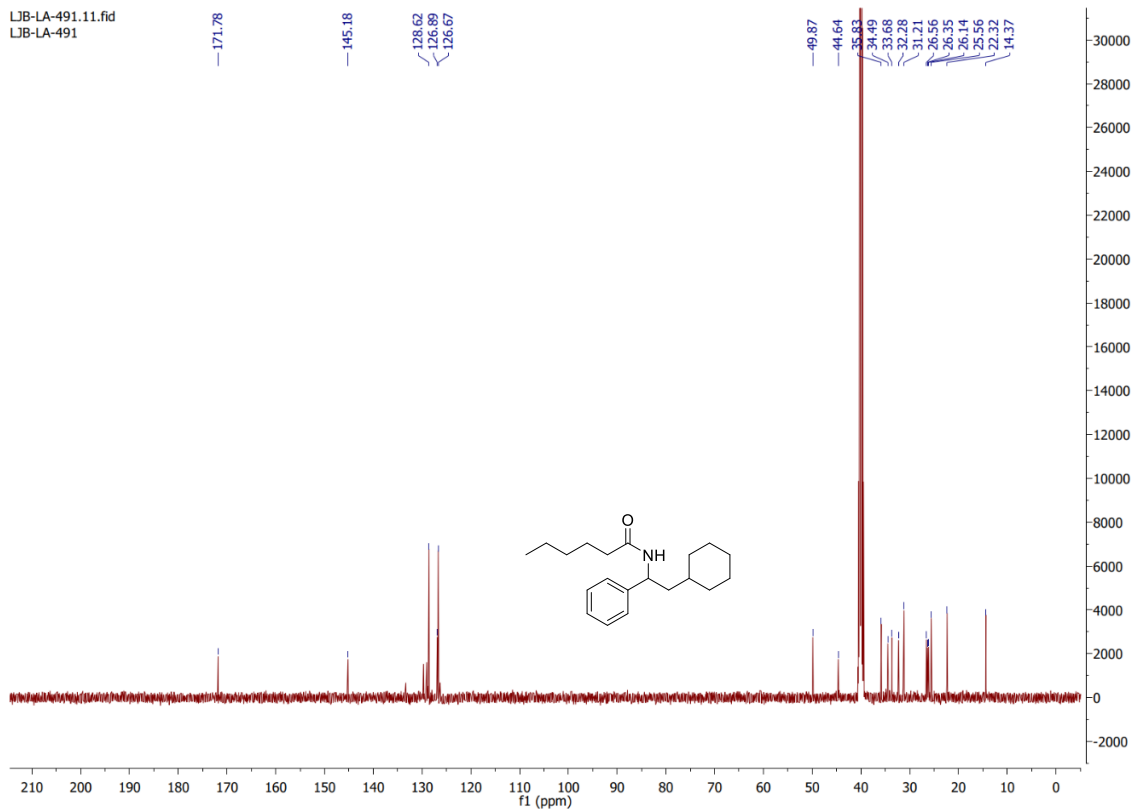




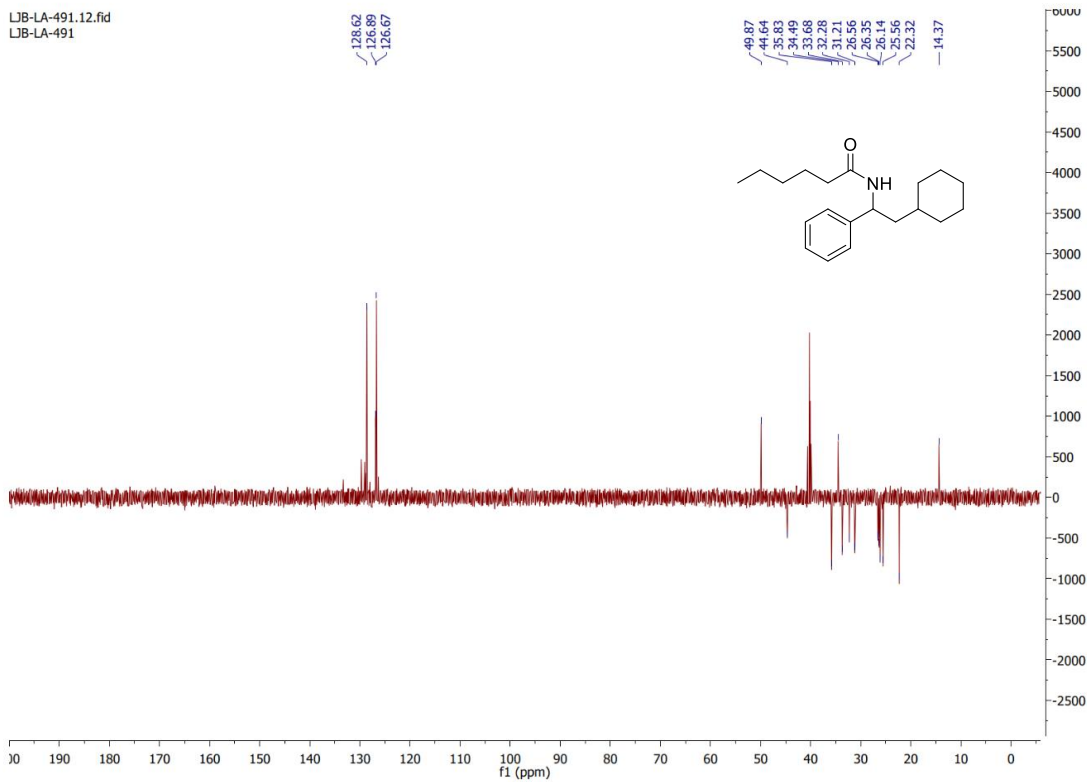
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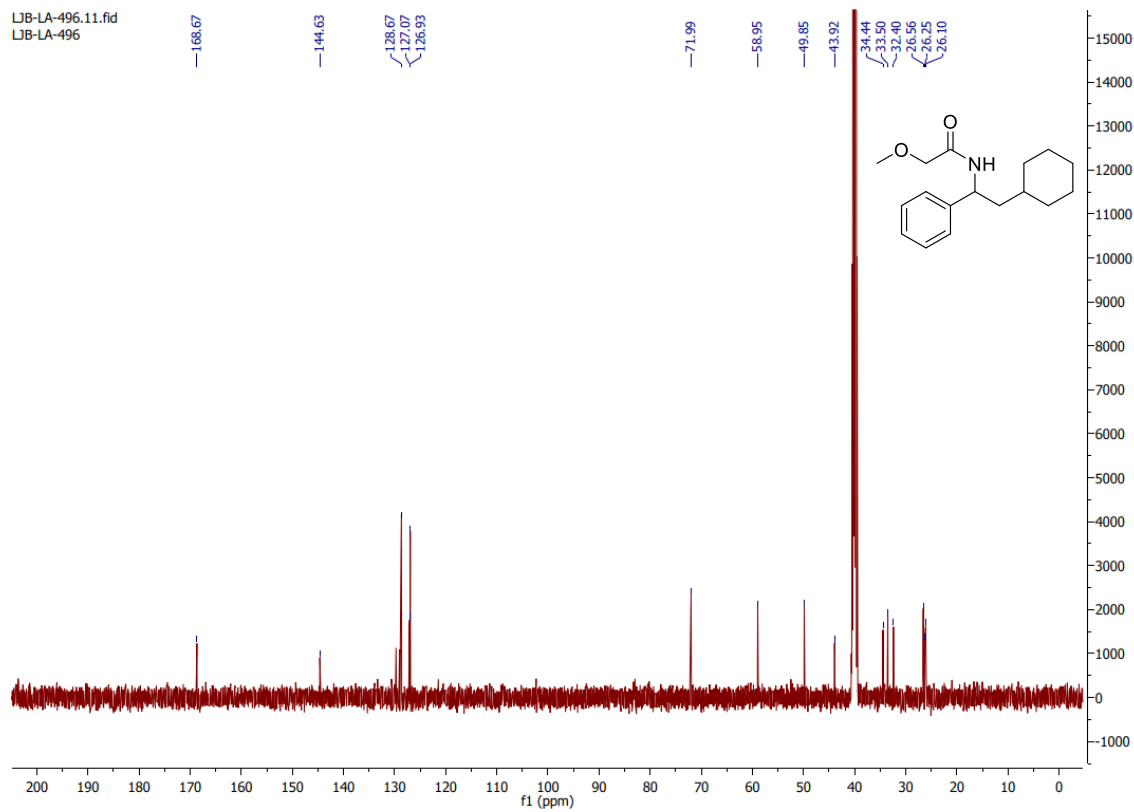
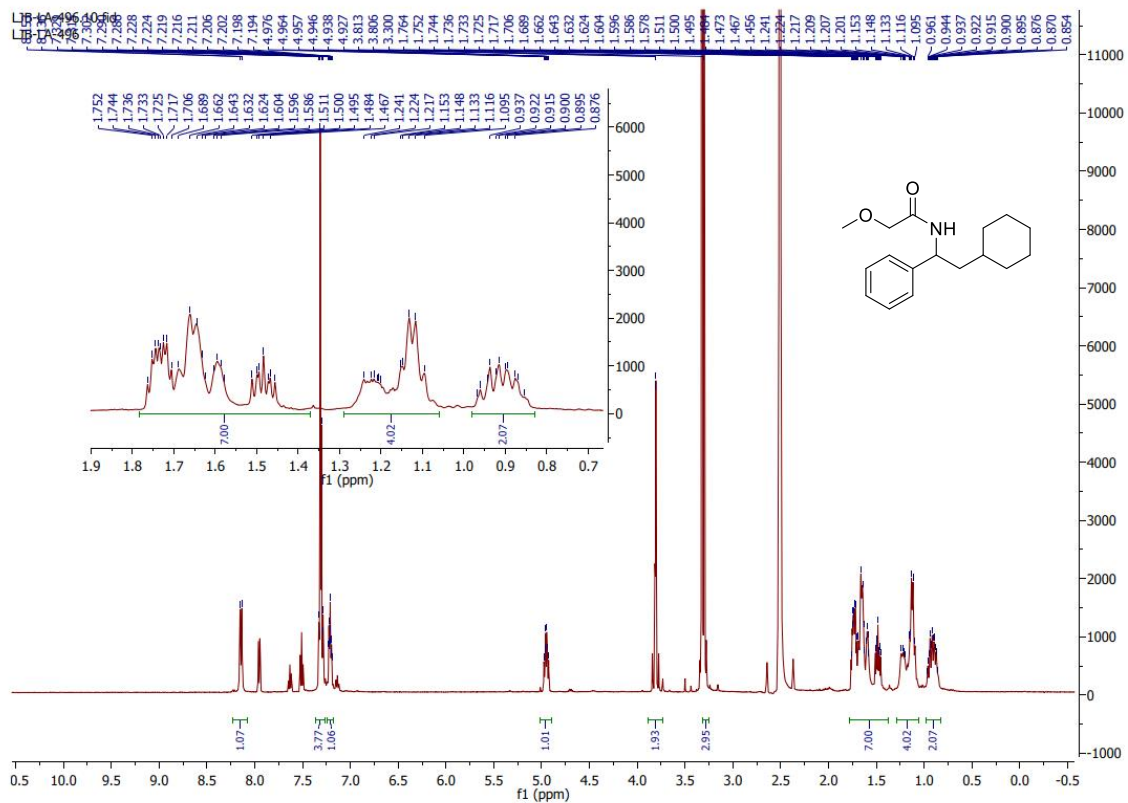


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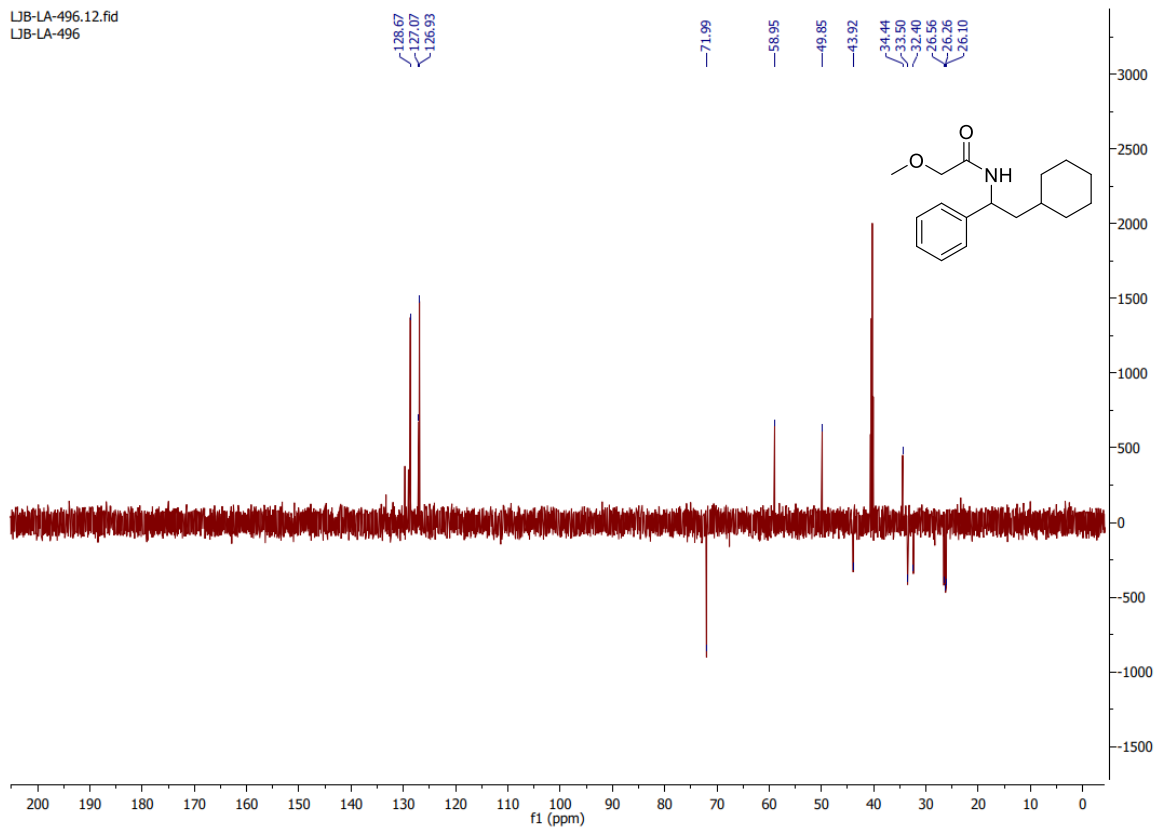


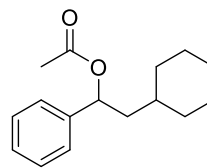
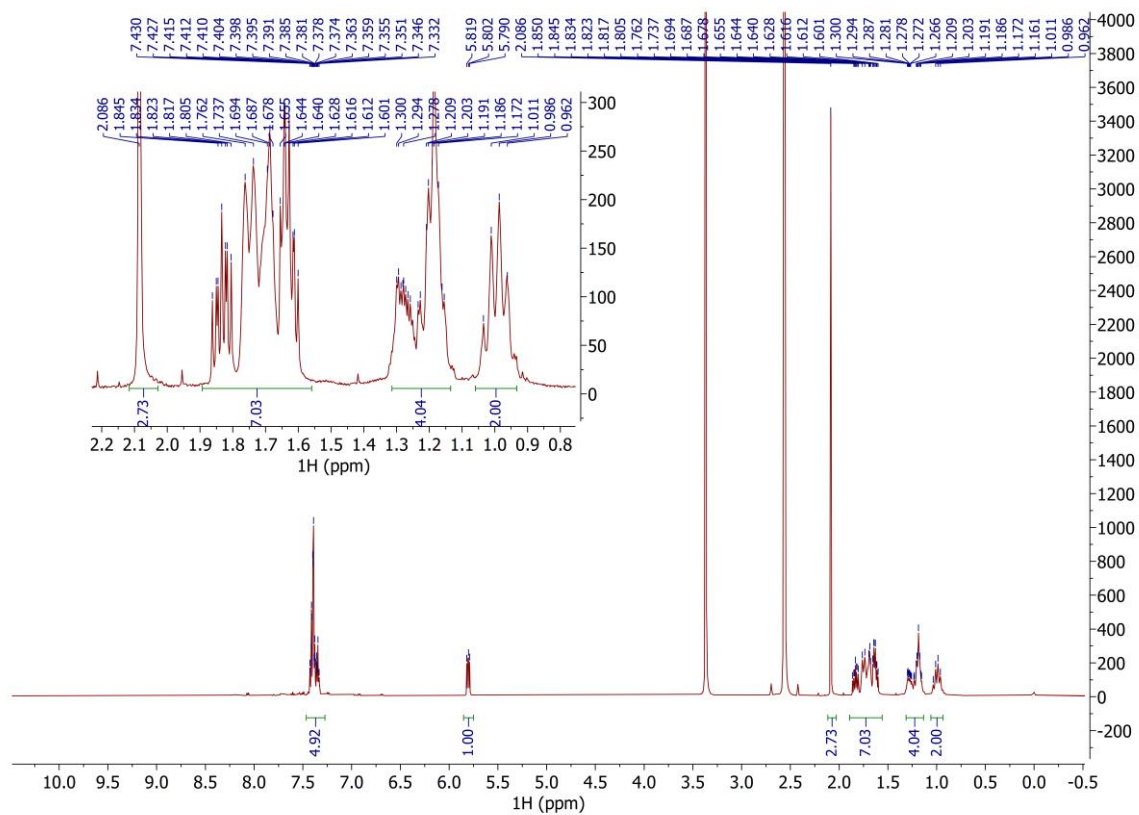
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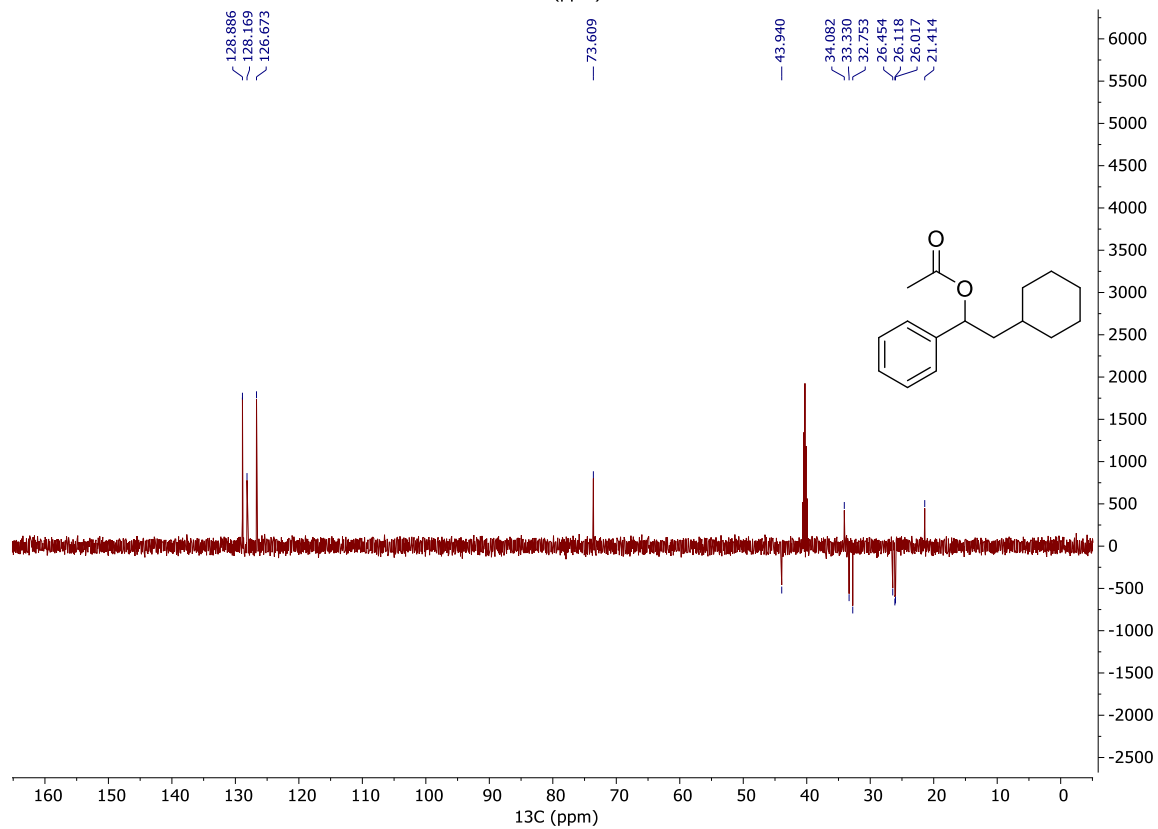
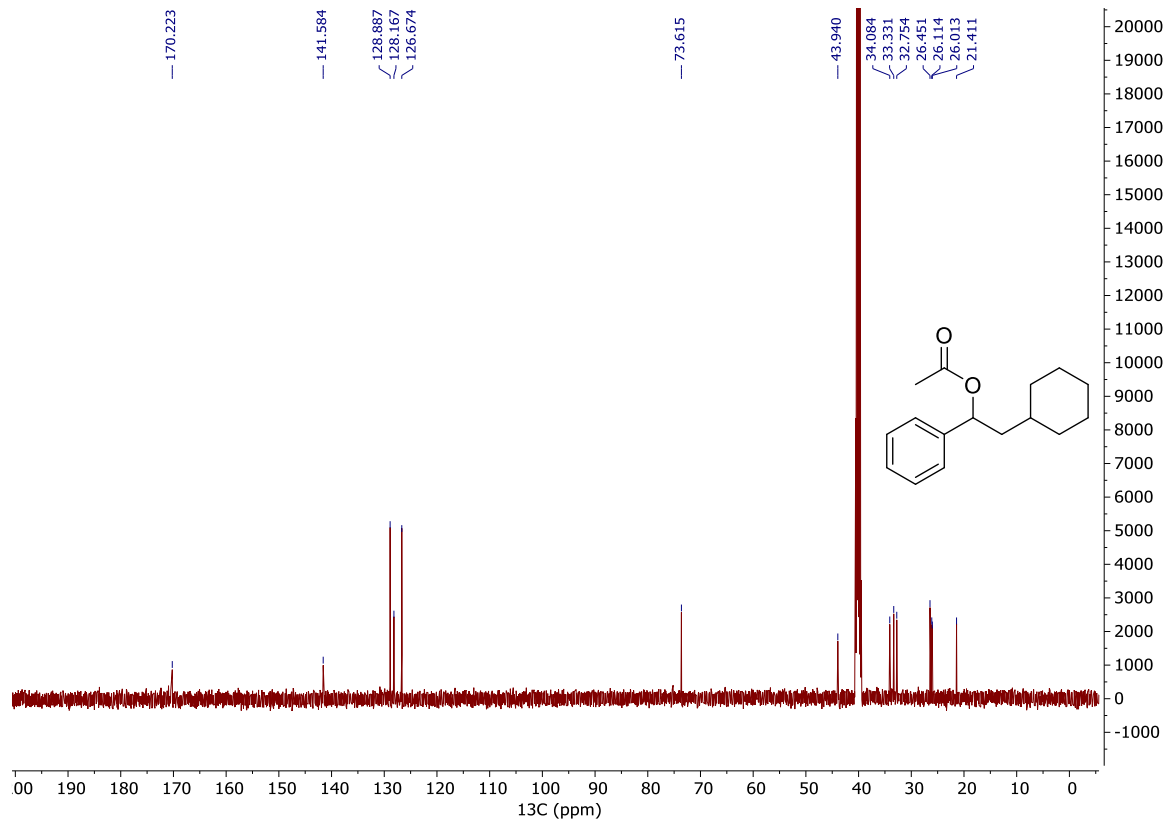




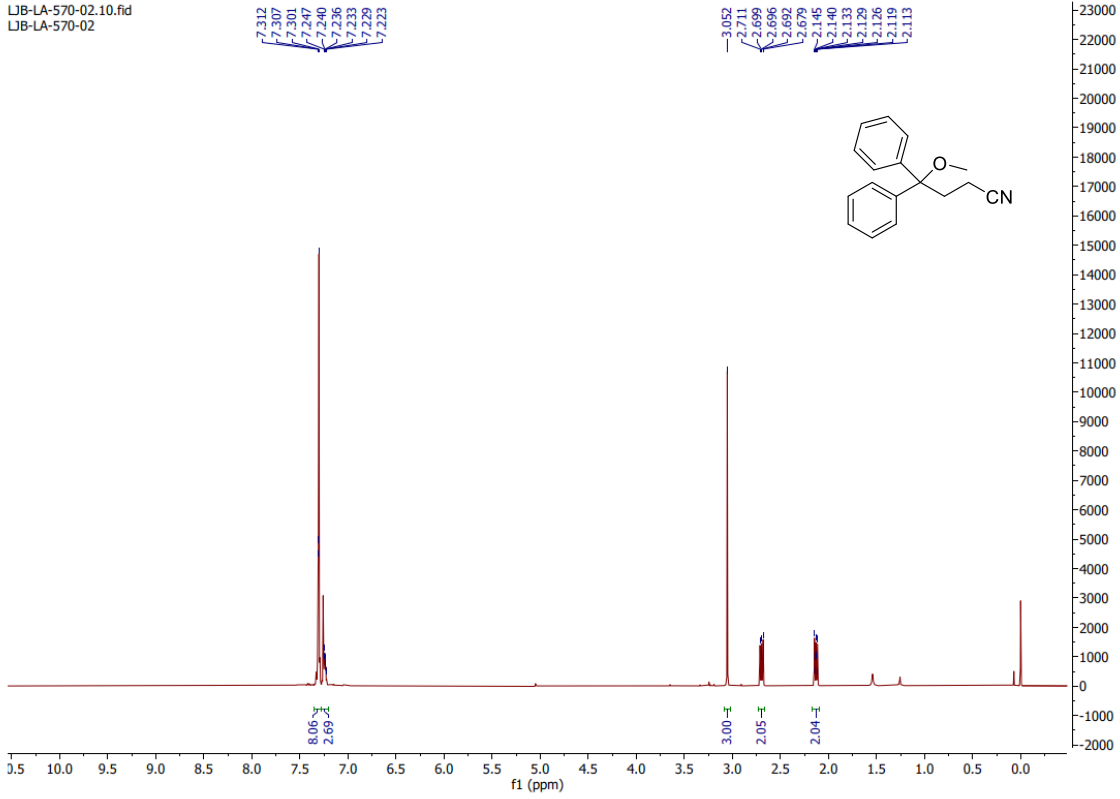
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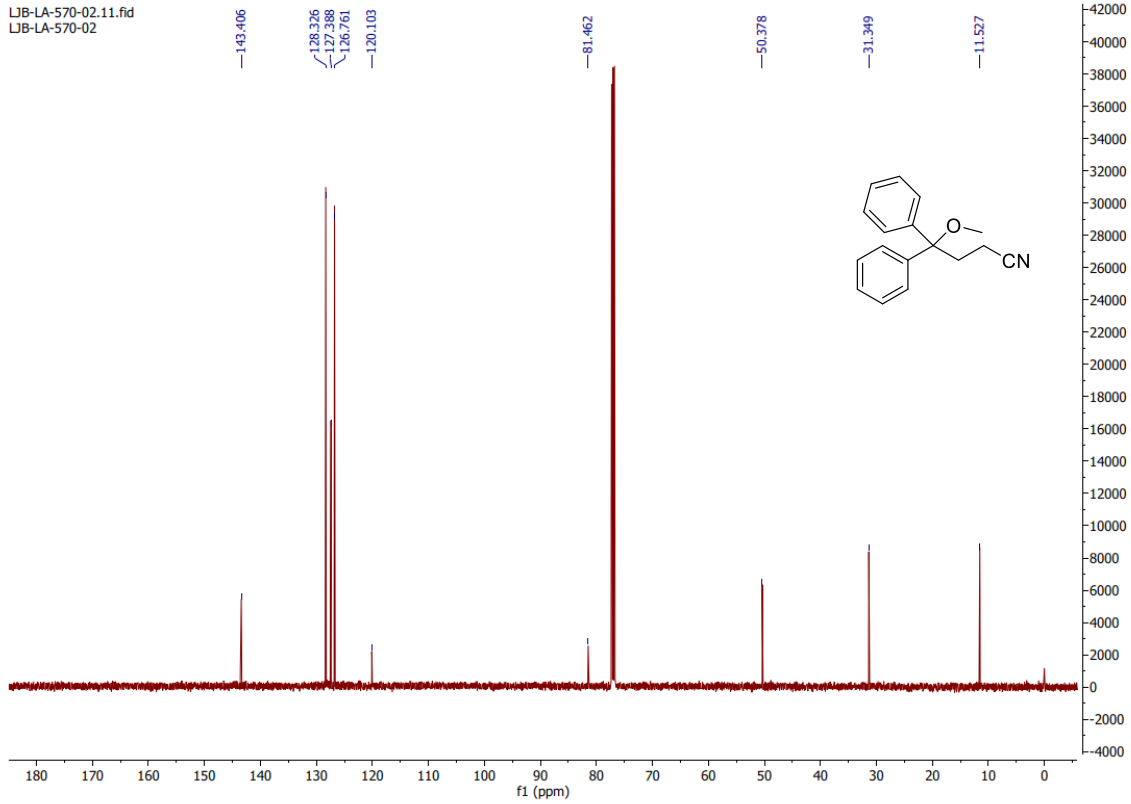




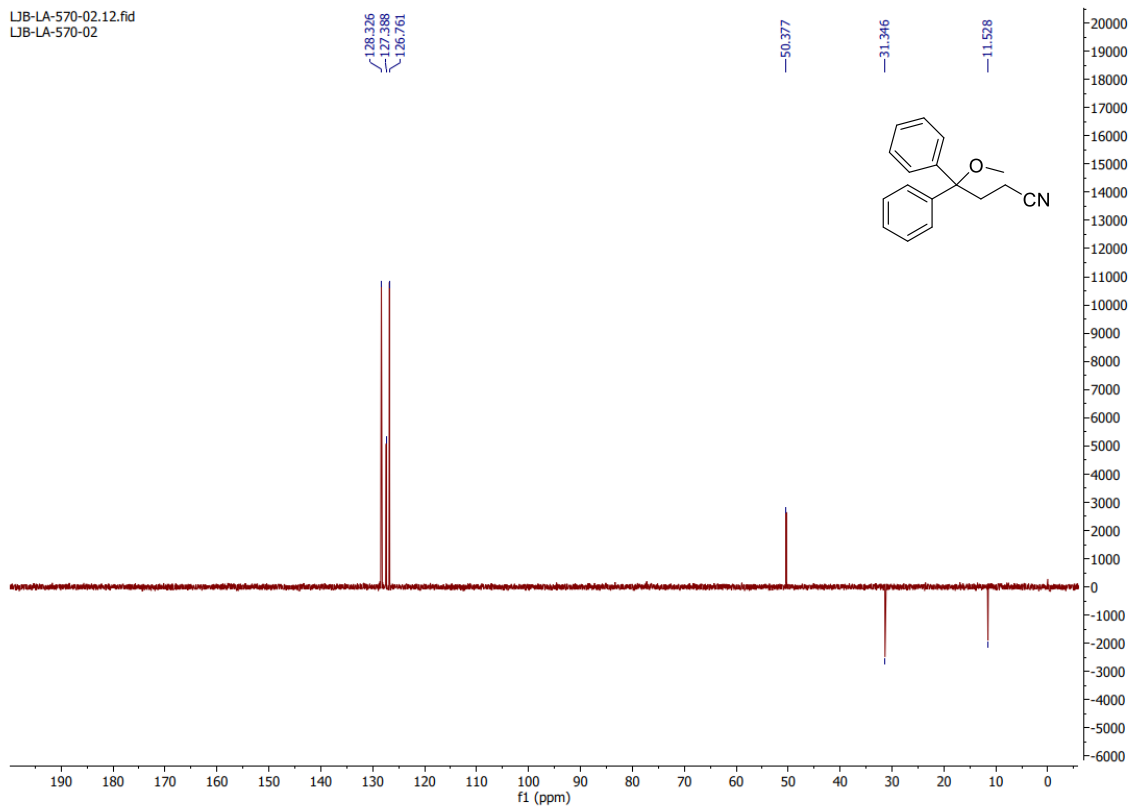
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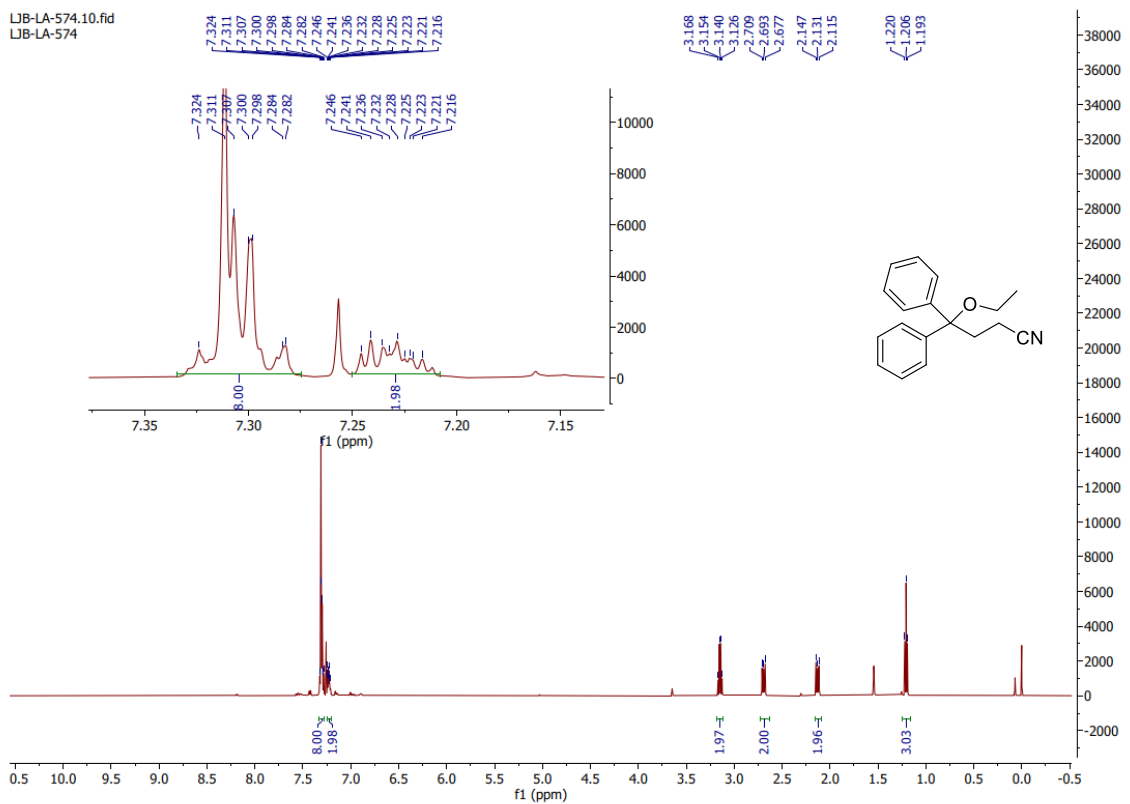
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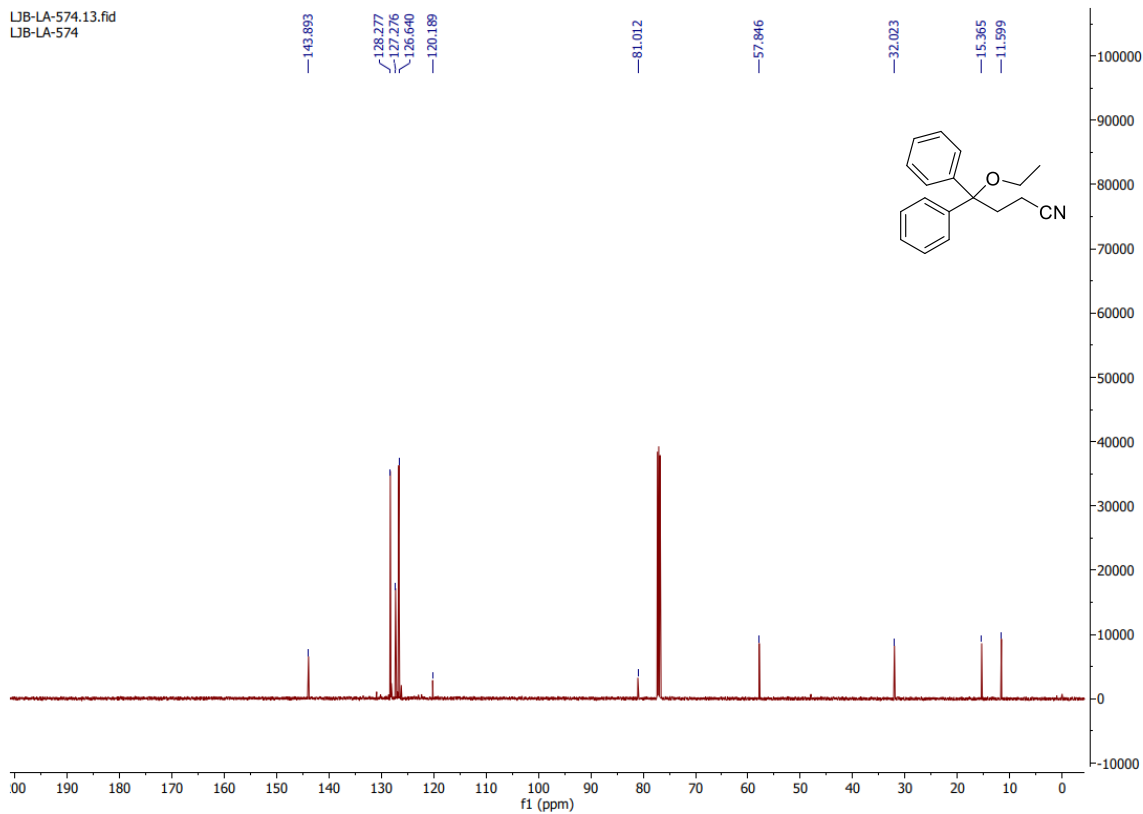
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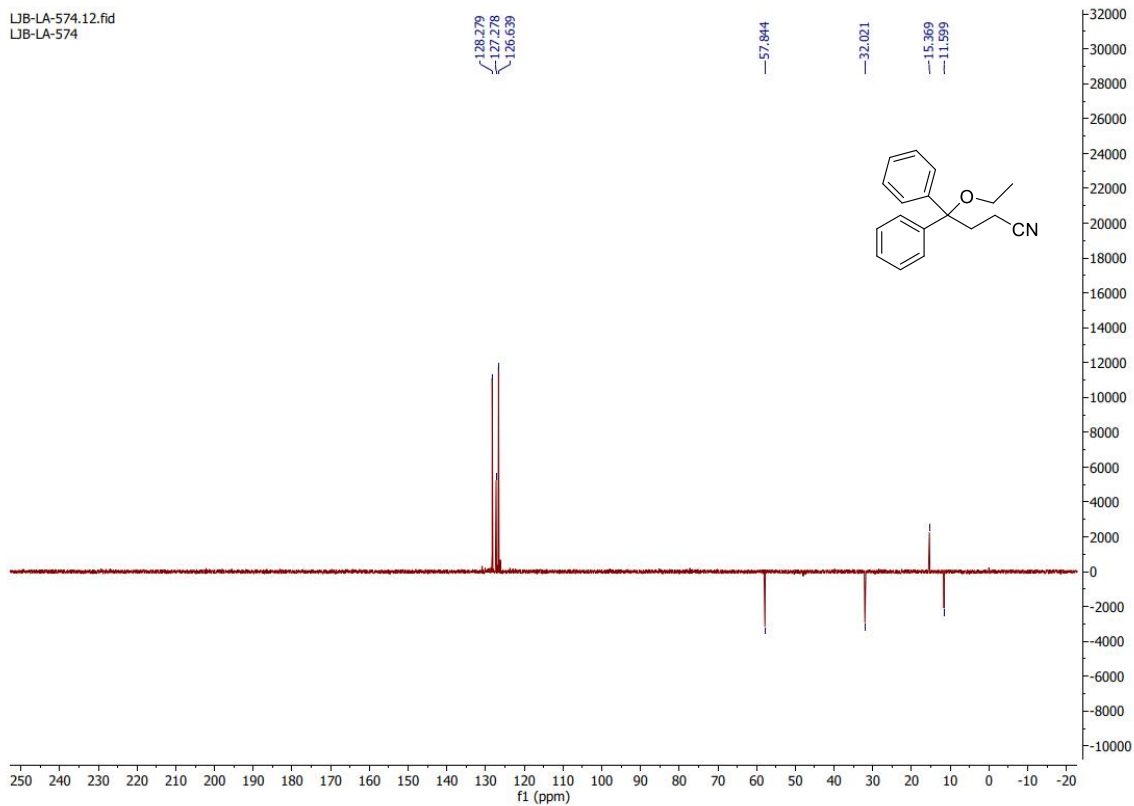
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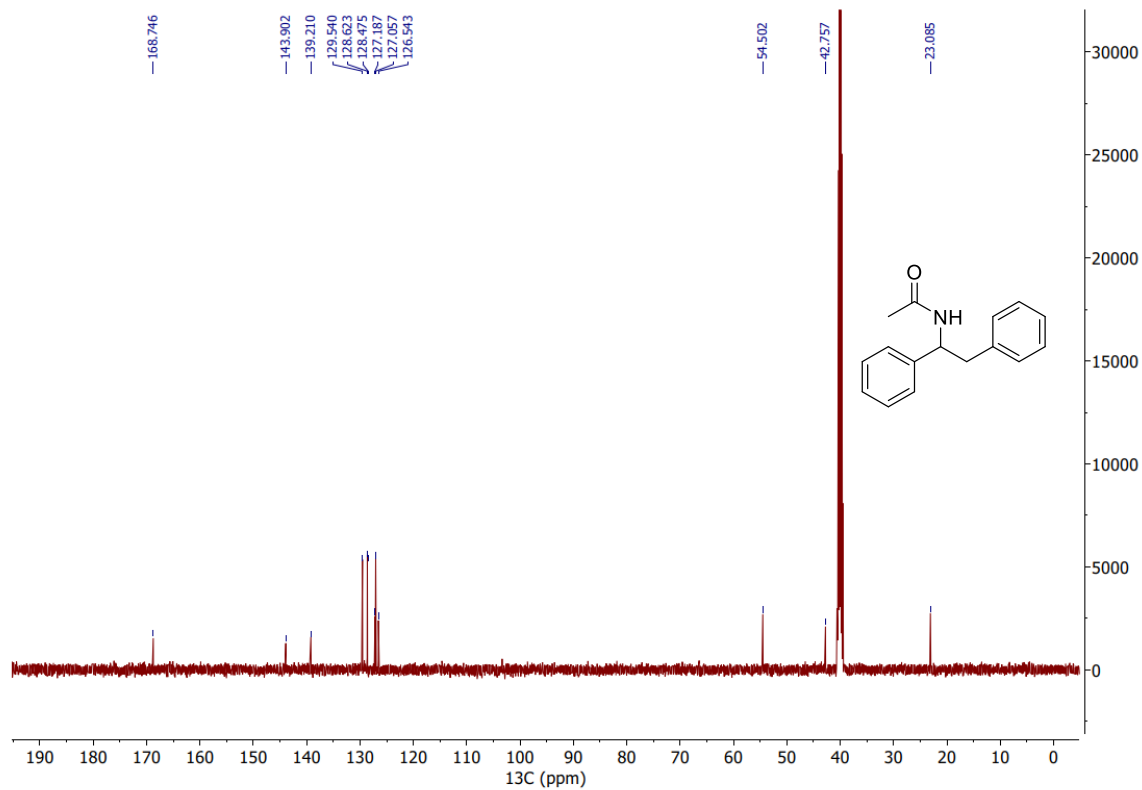
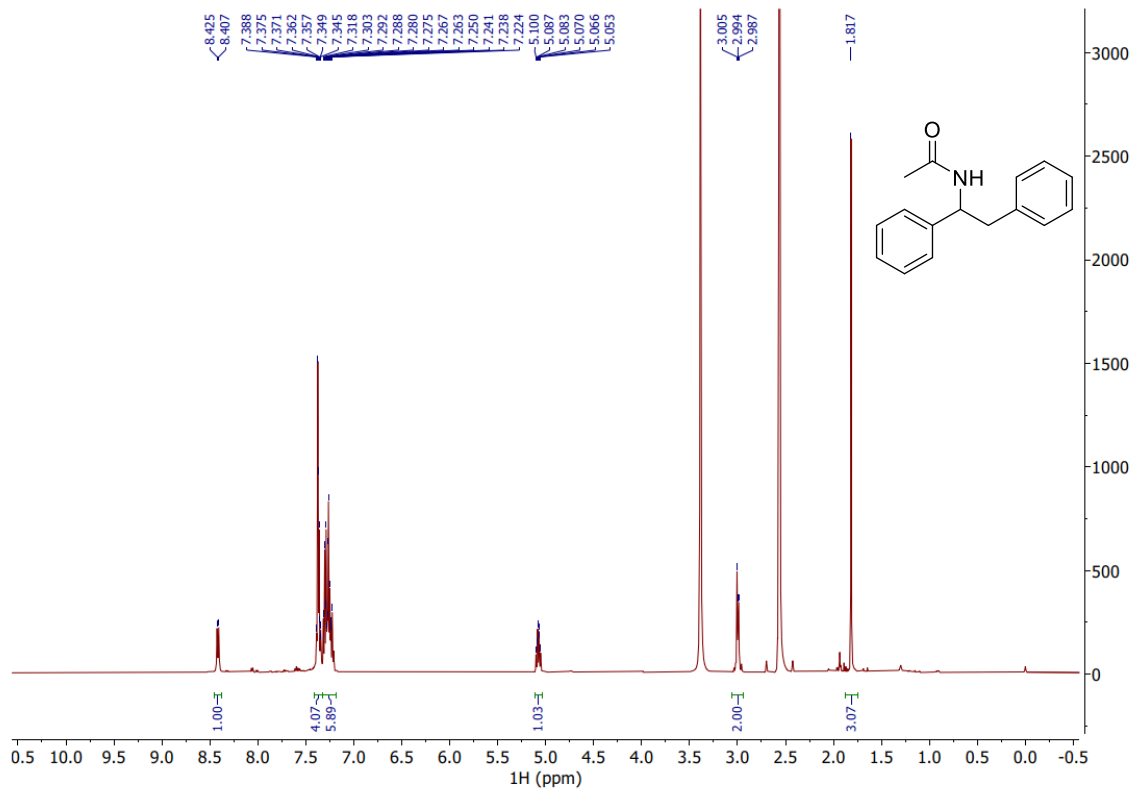


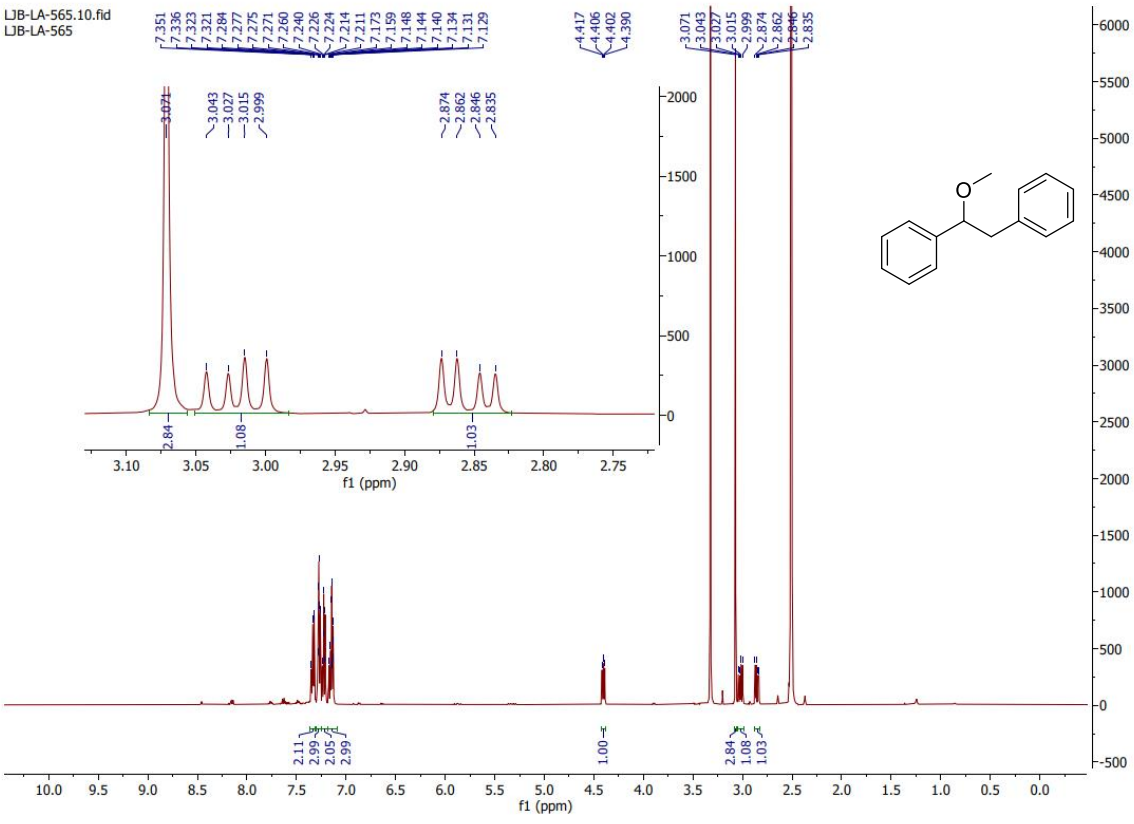
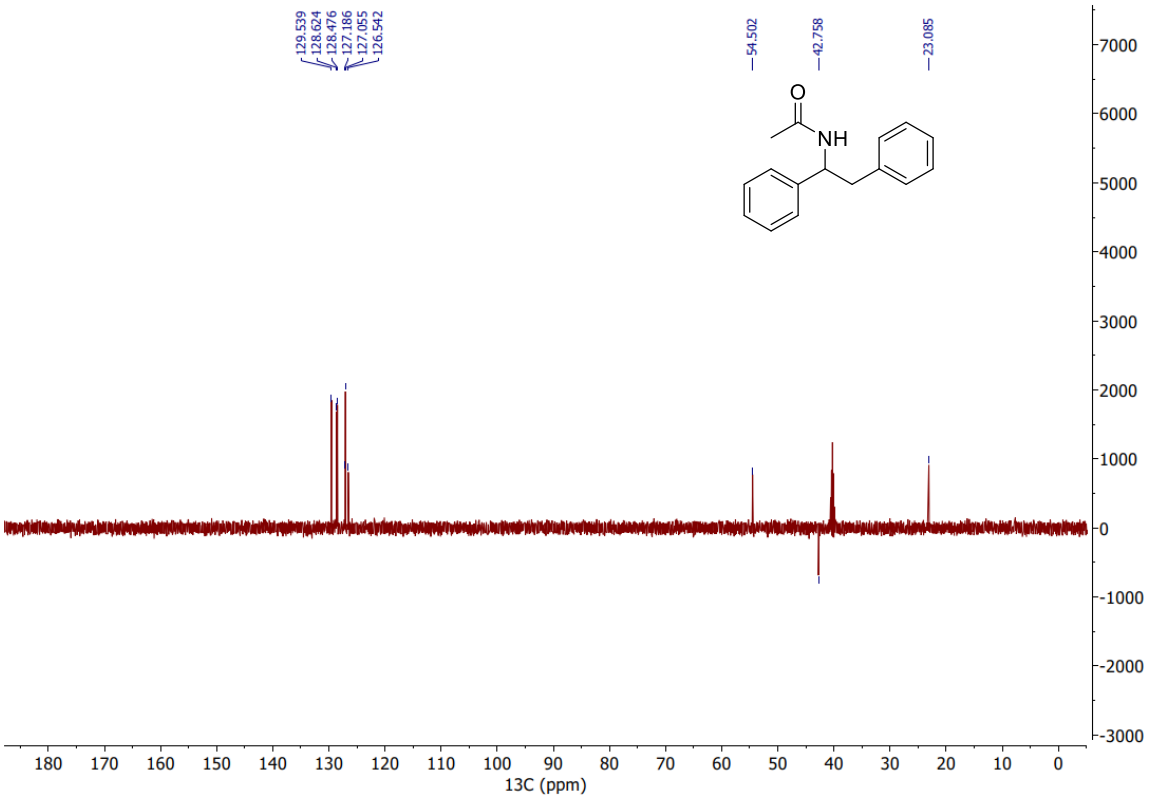
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LJB-LA-574



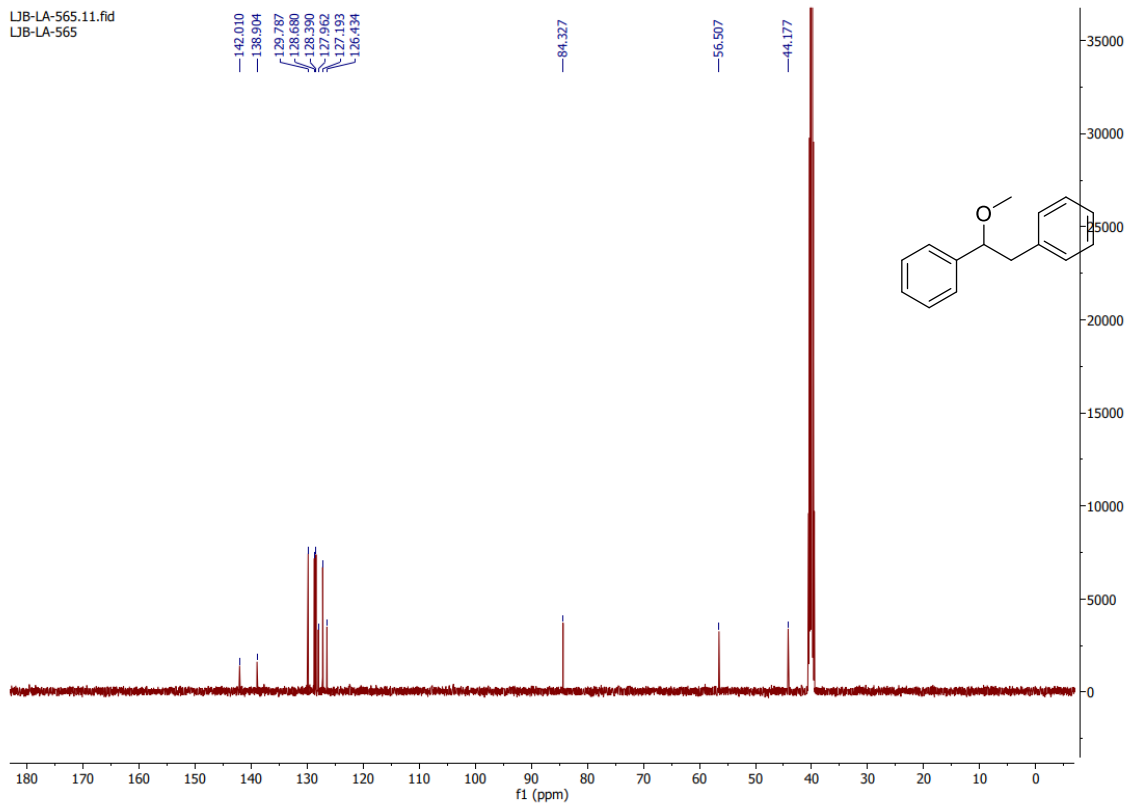
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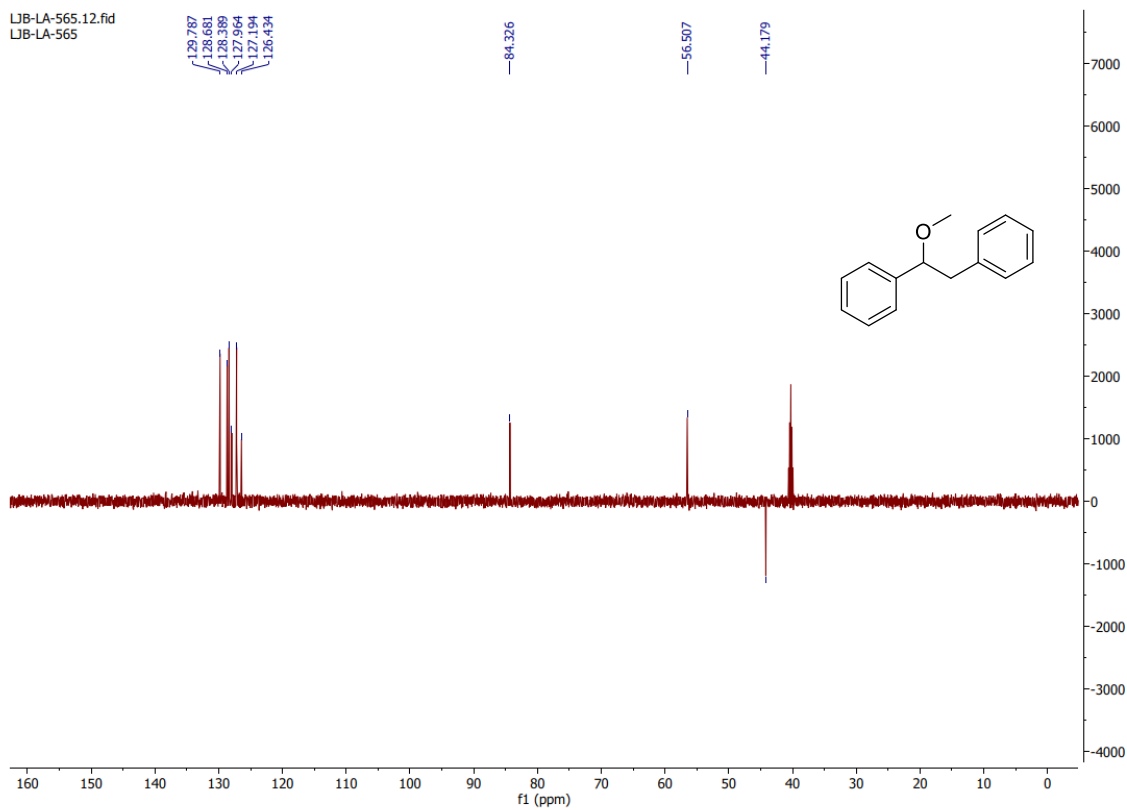




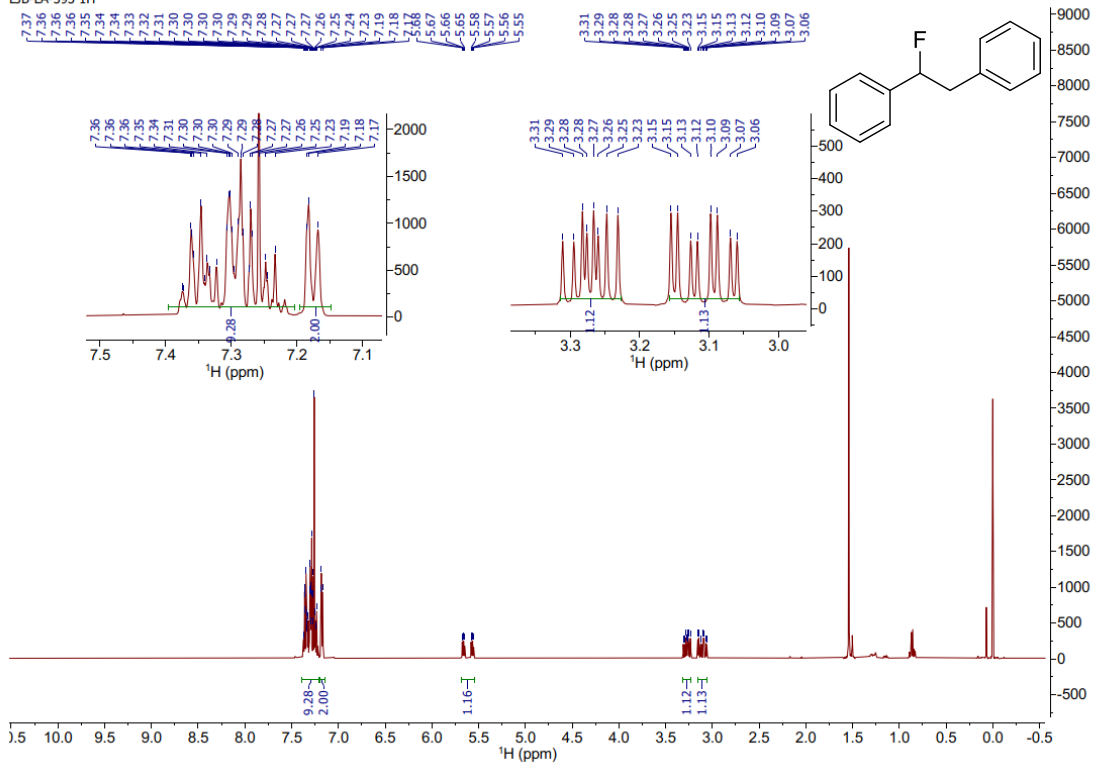
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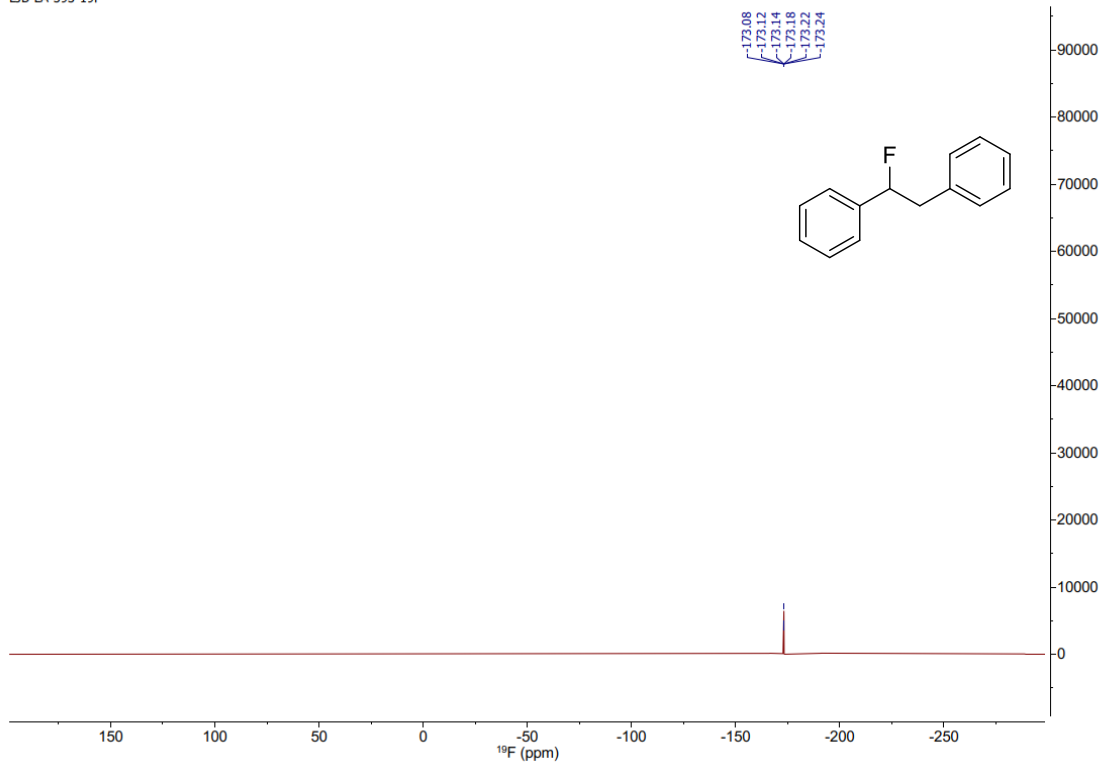
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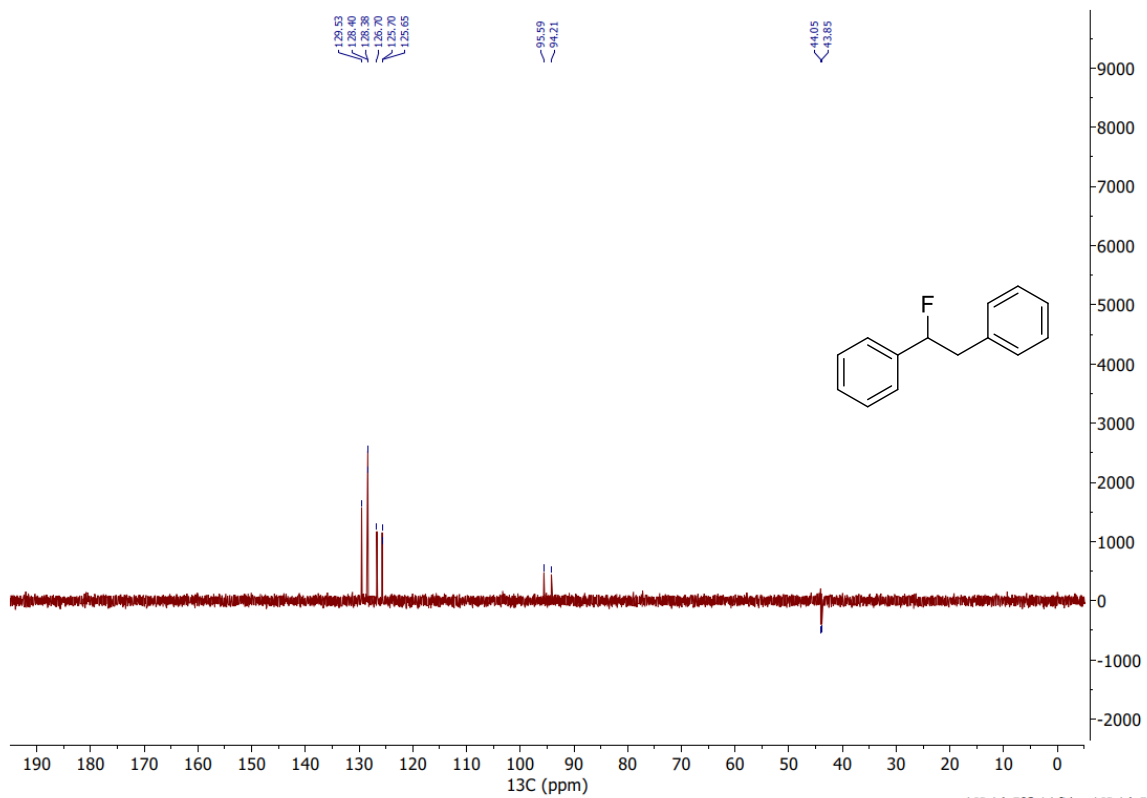
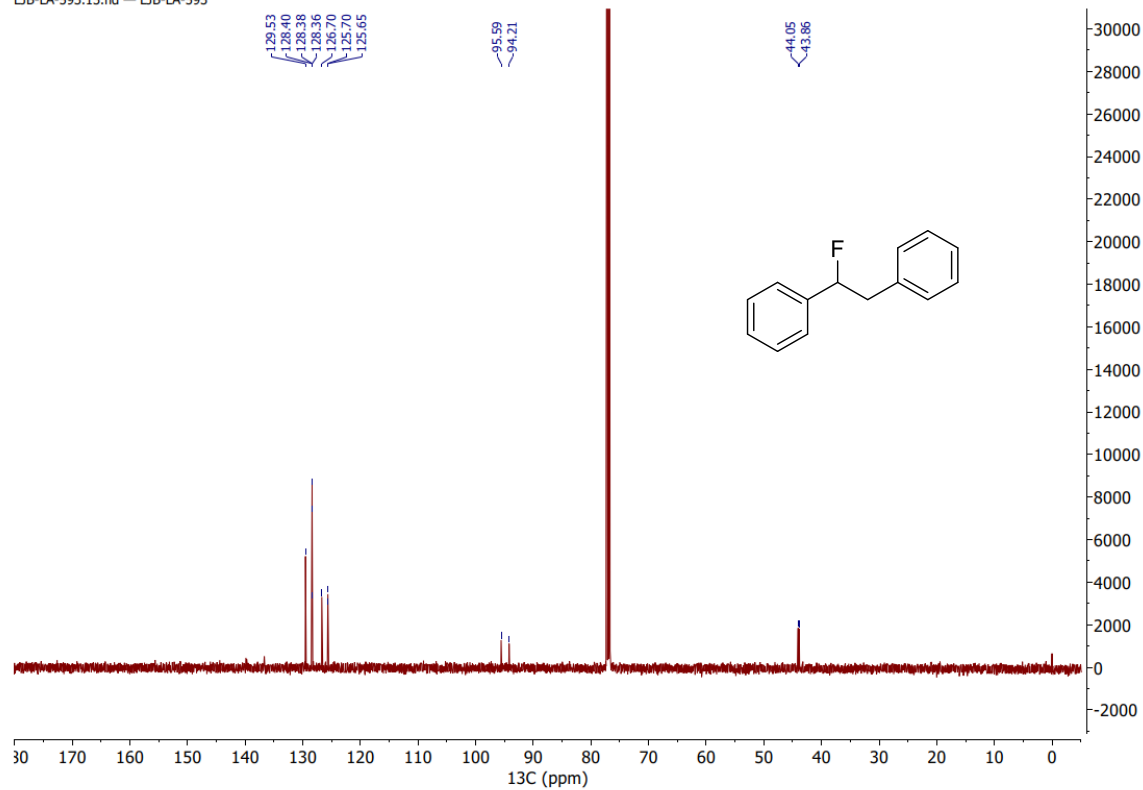
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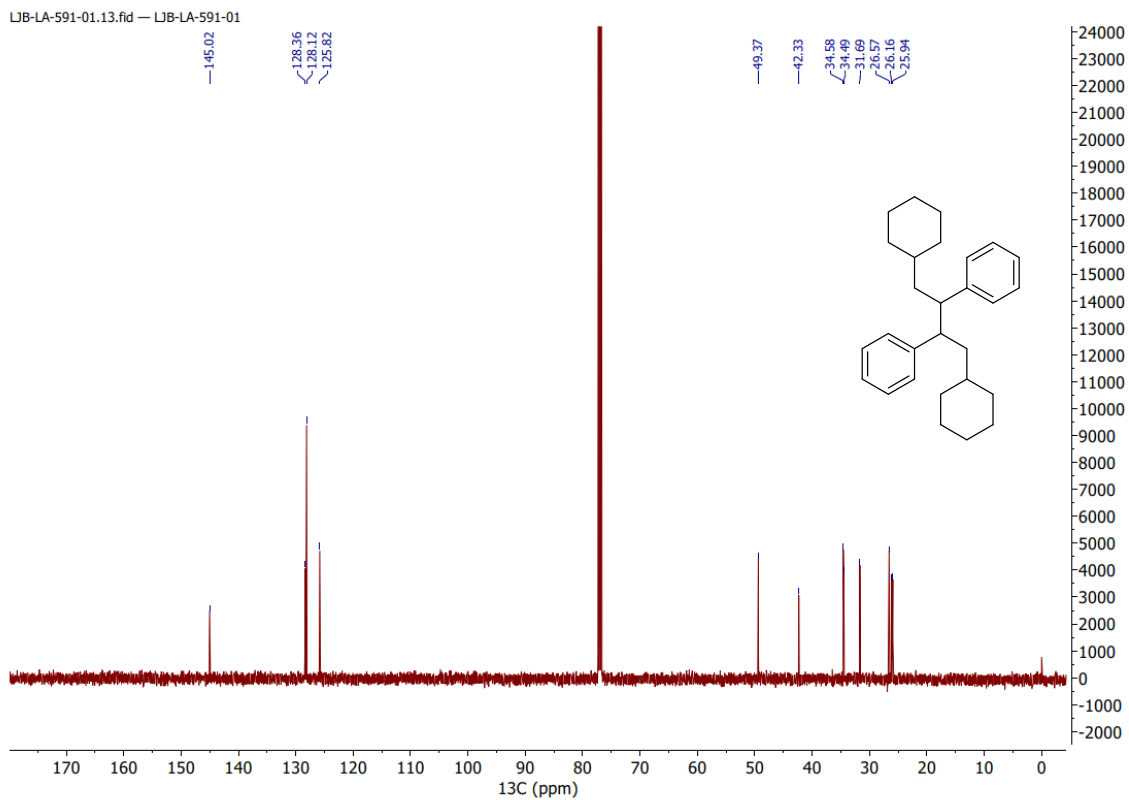
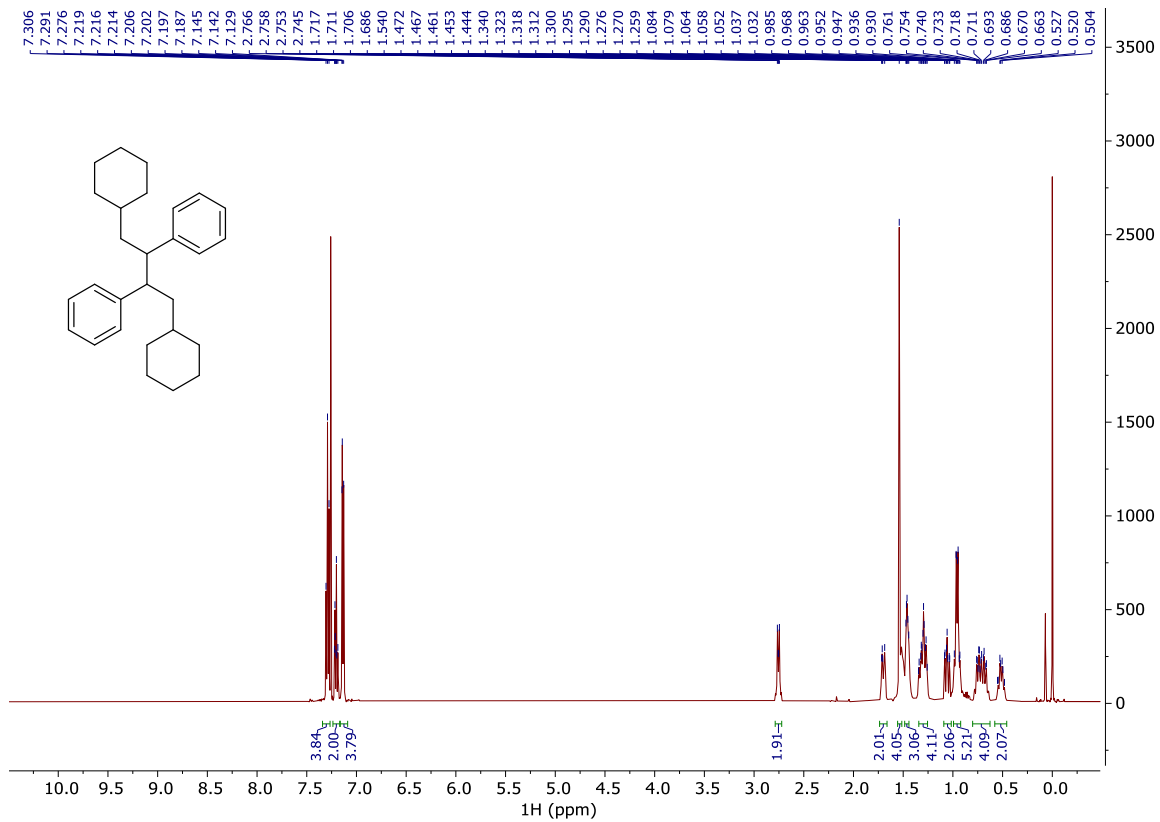
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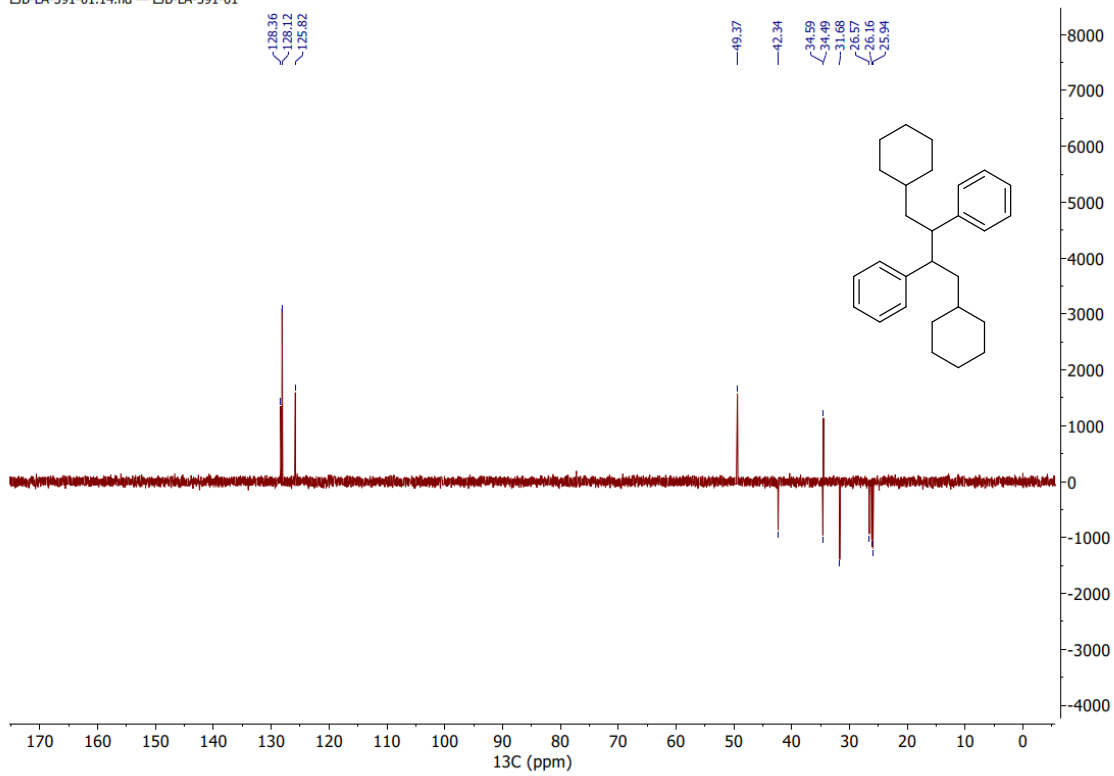
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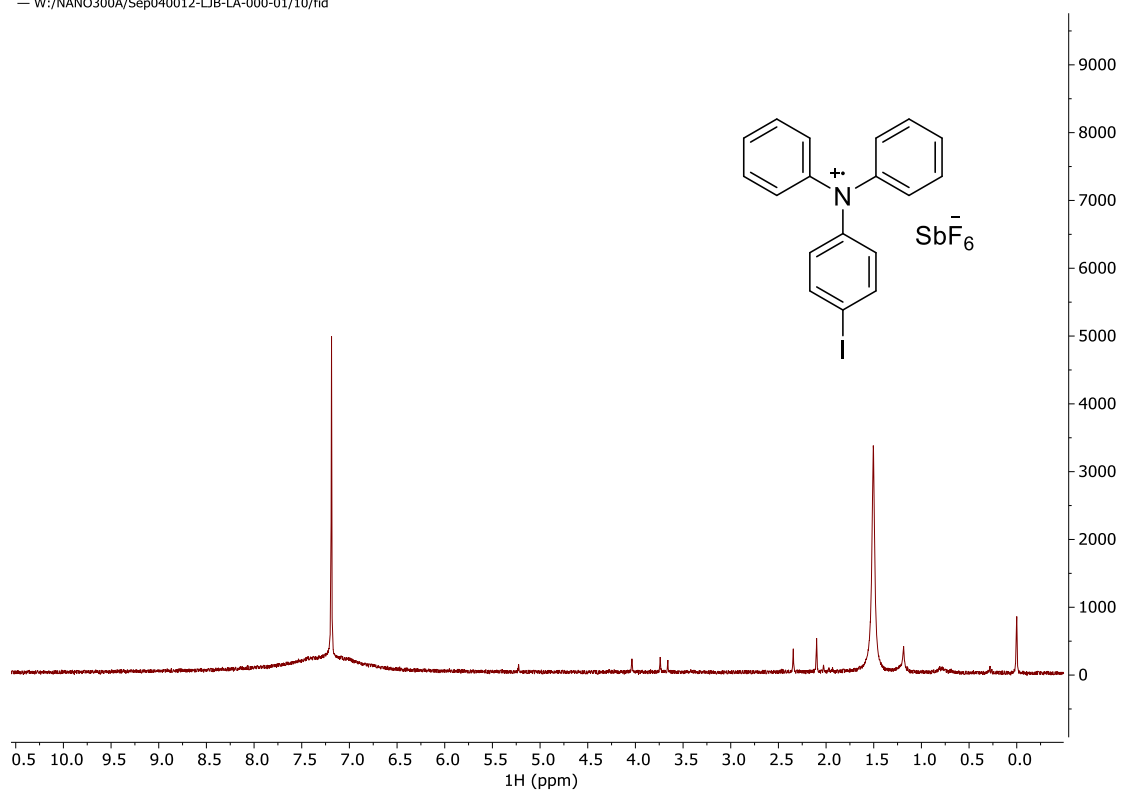
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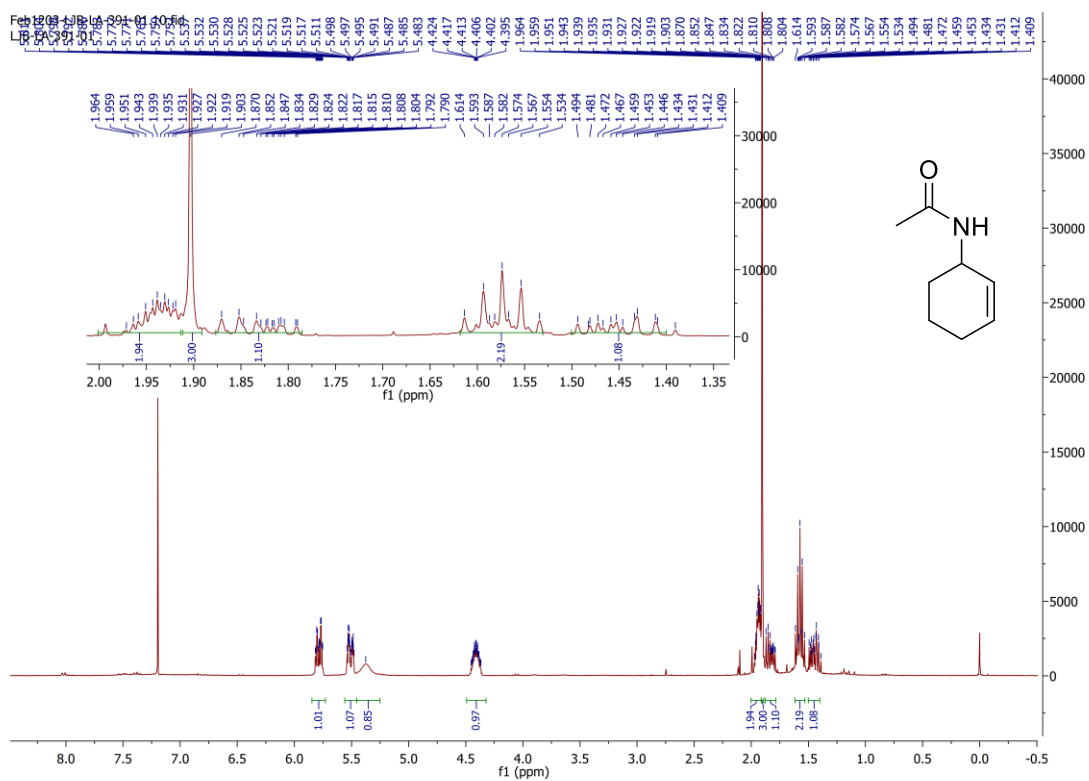
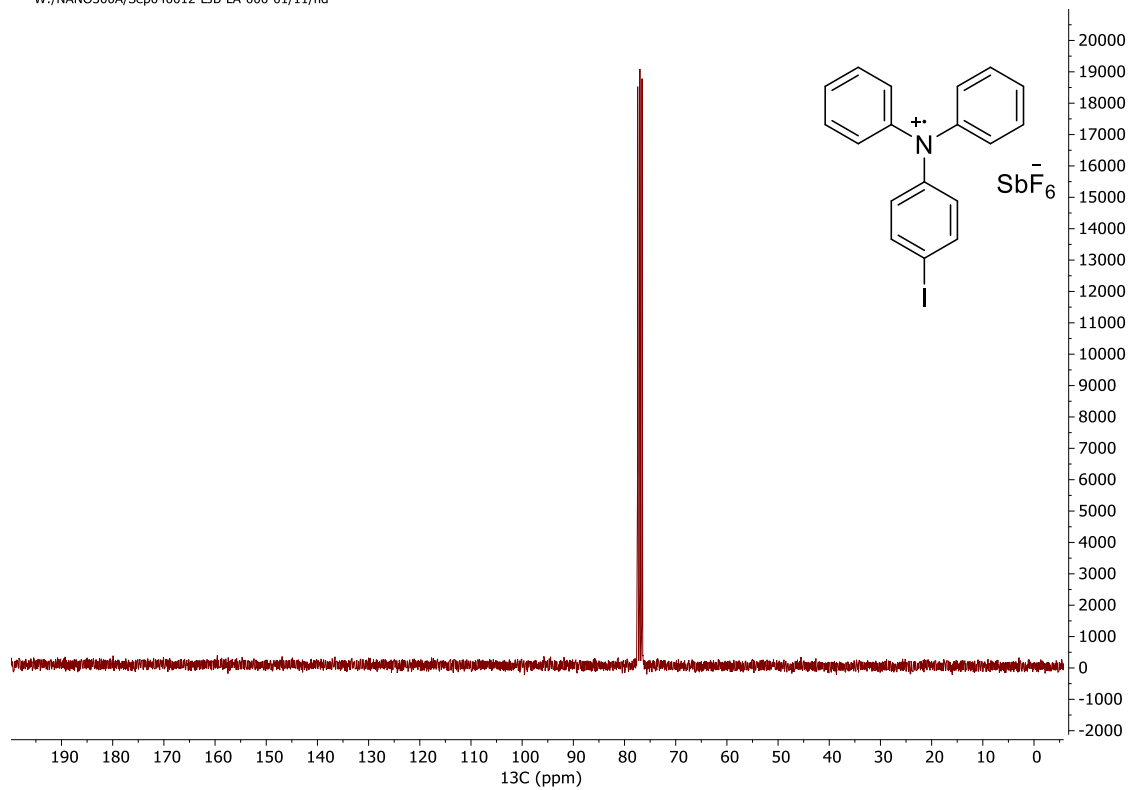


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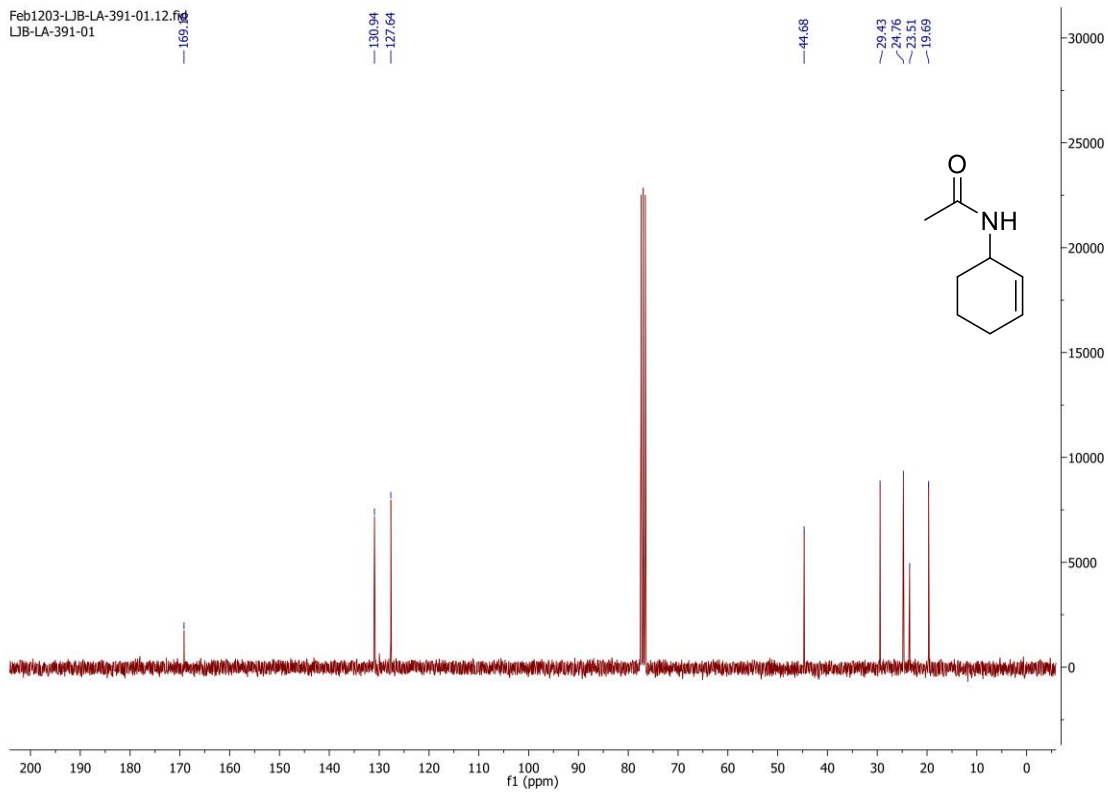


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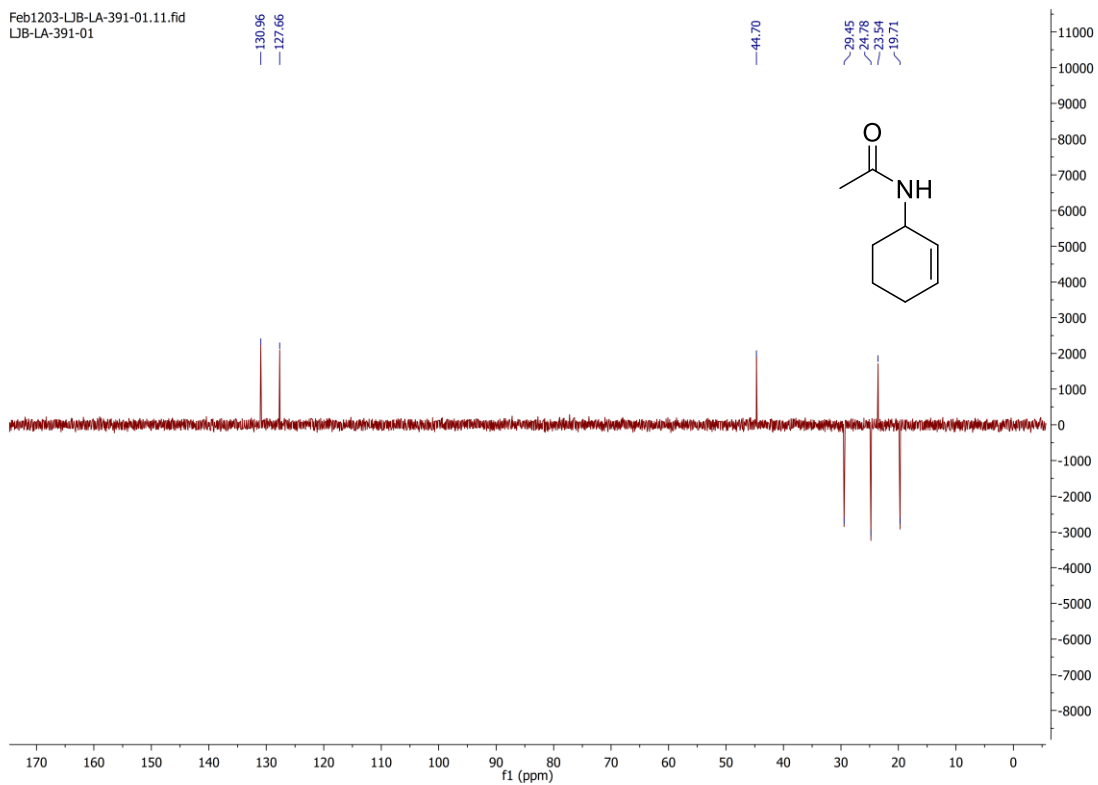




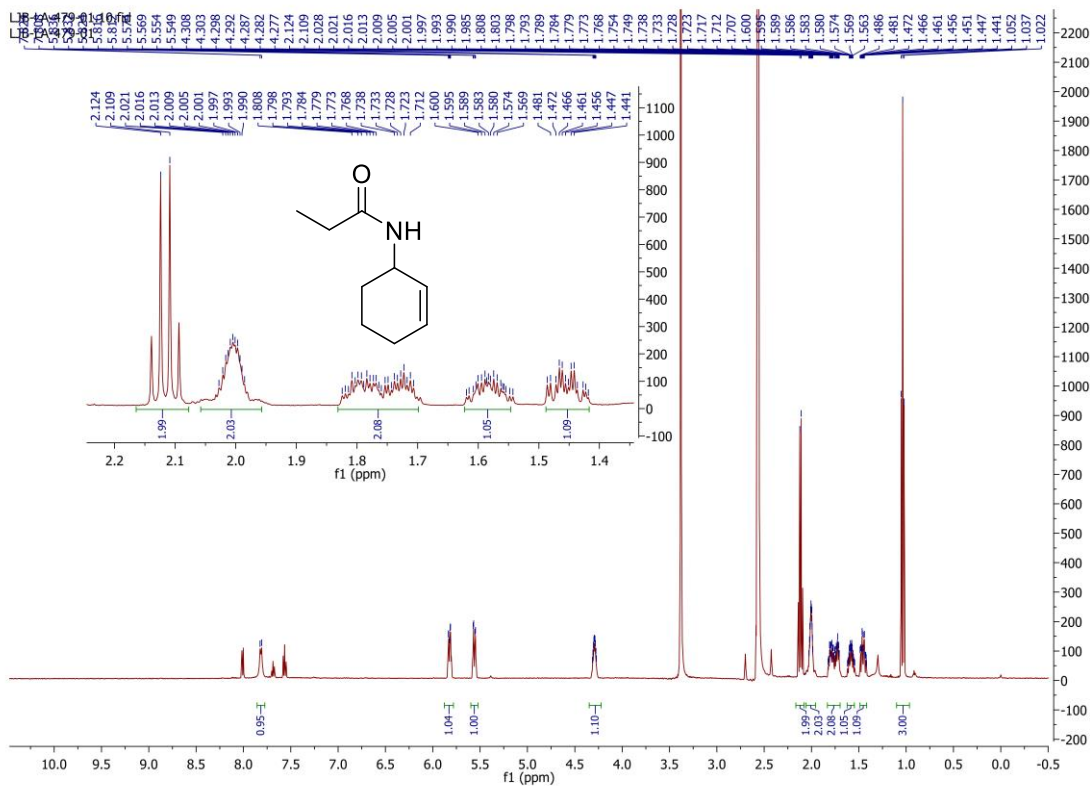
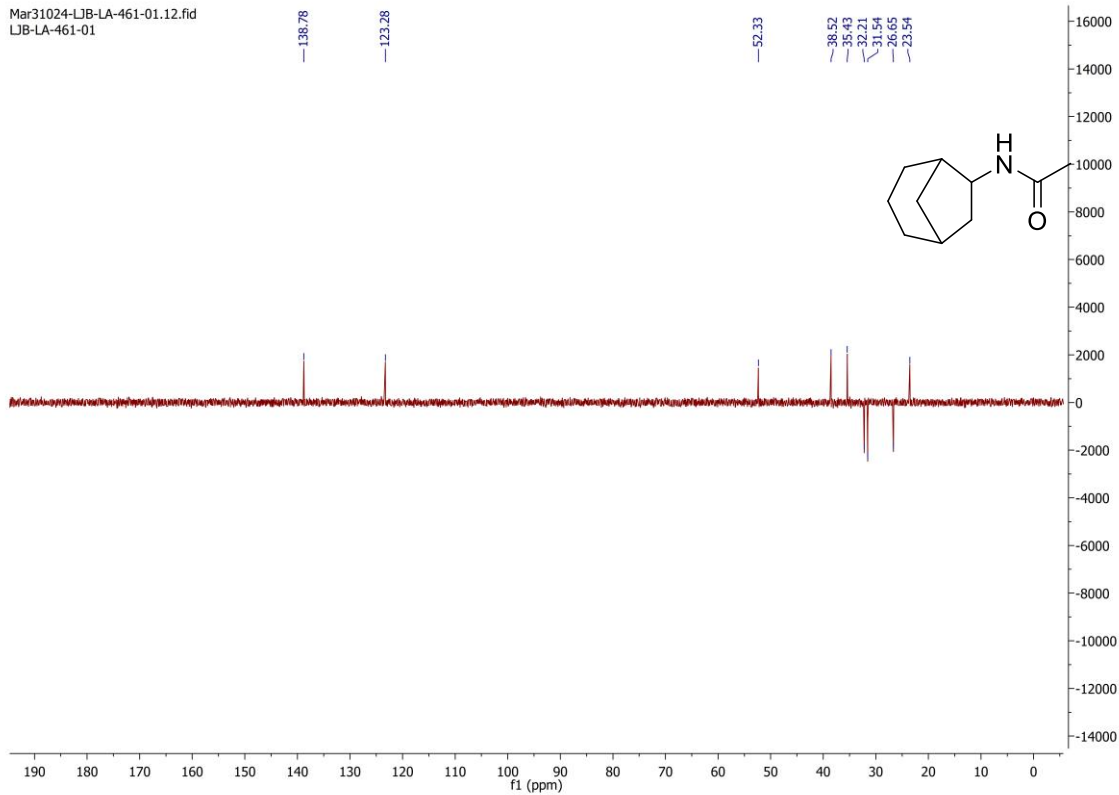
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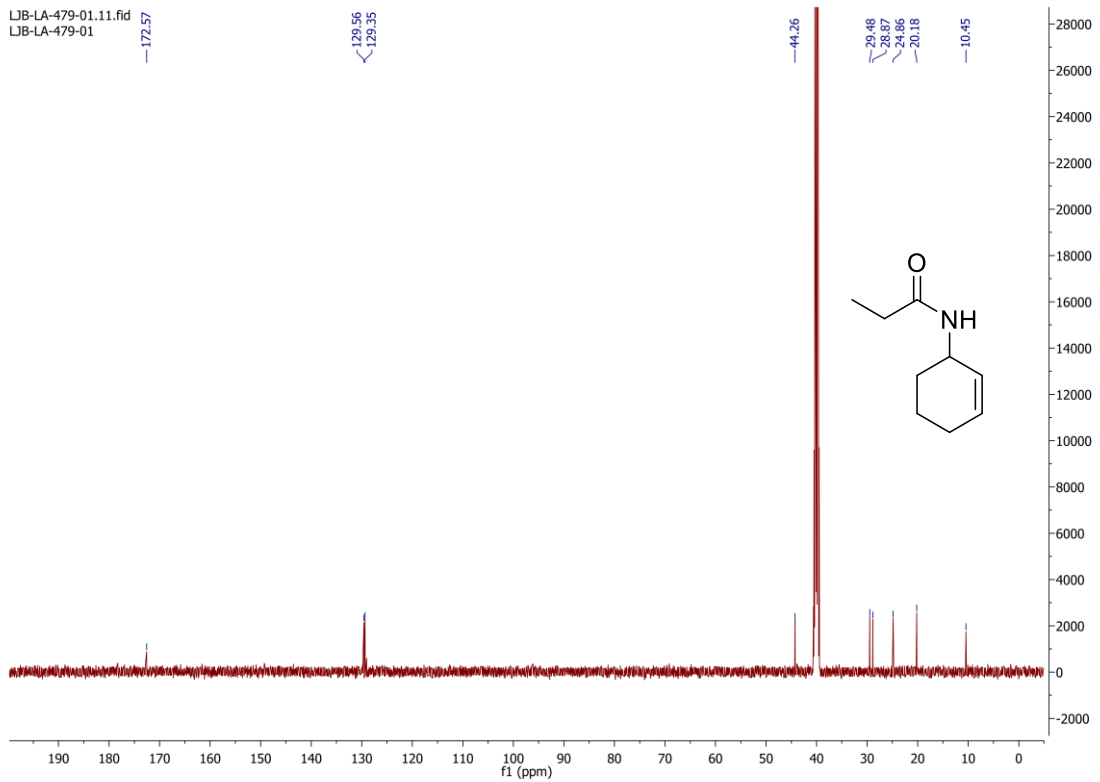
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LJB-LA-391-01



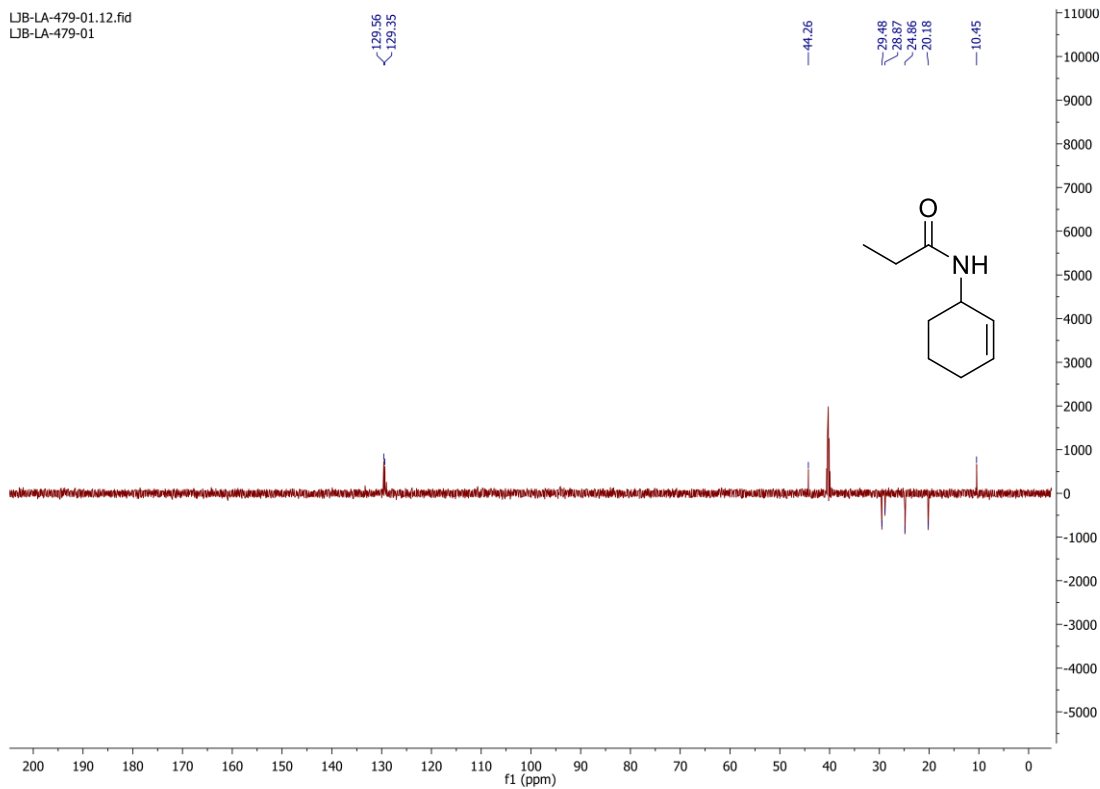
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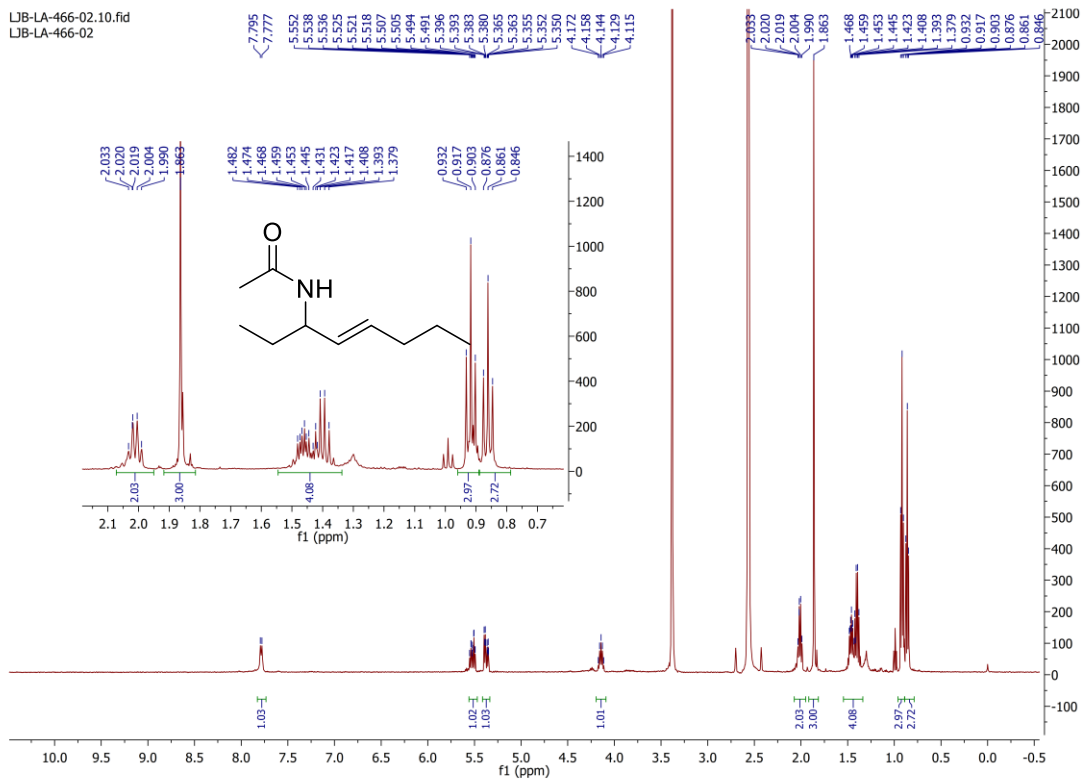
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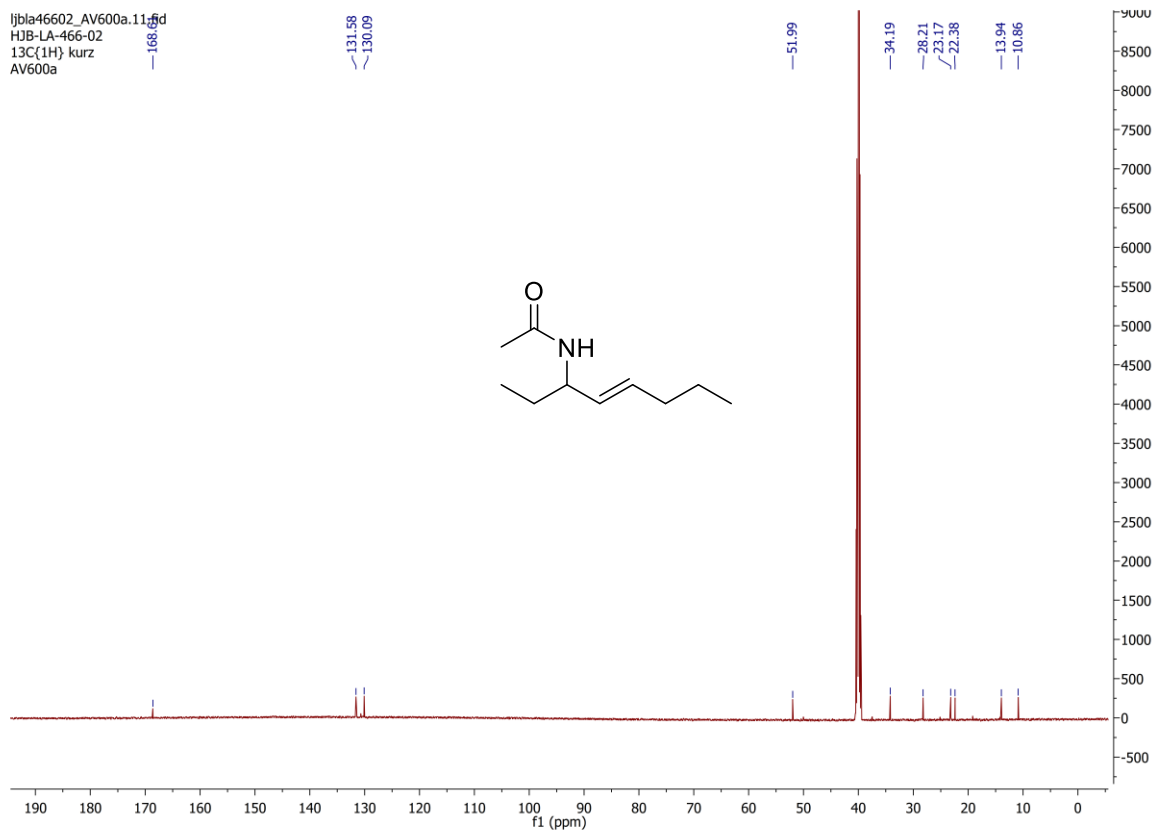
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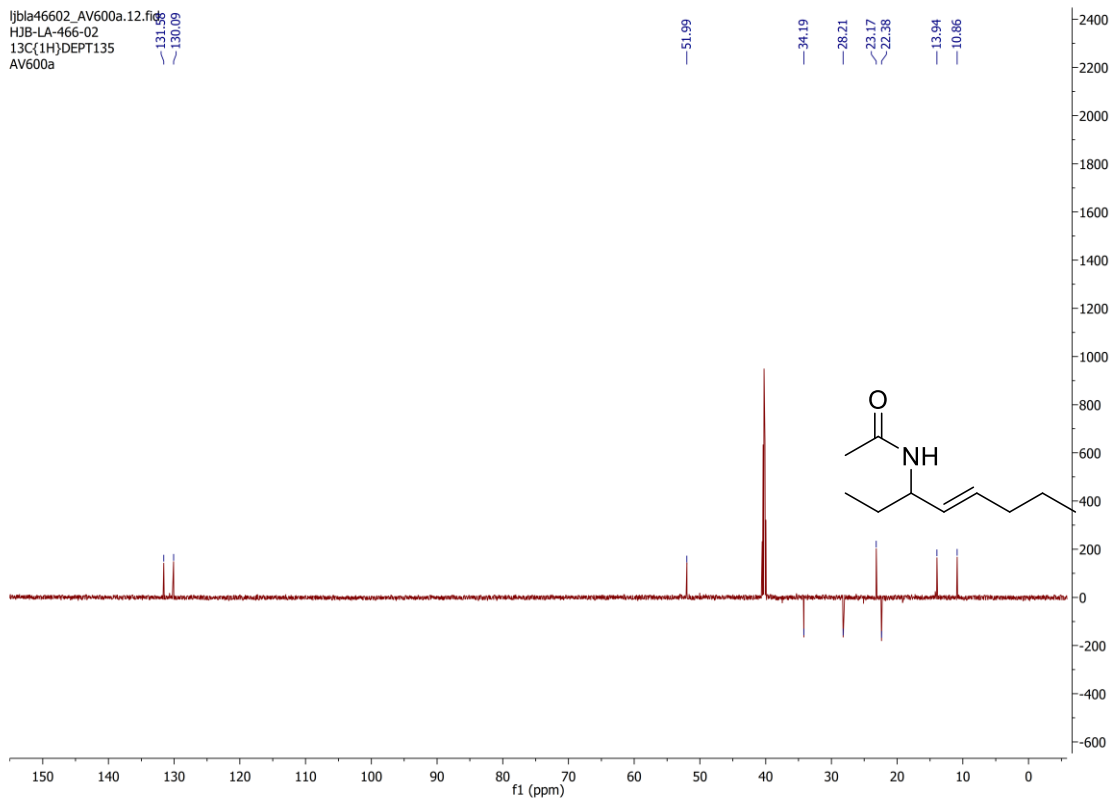
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LJB-LA-466-02



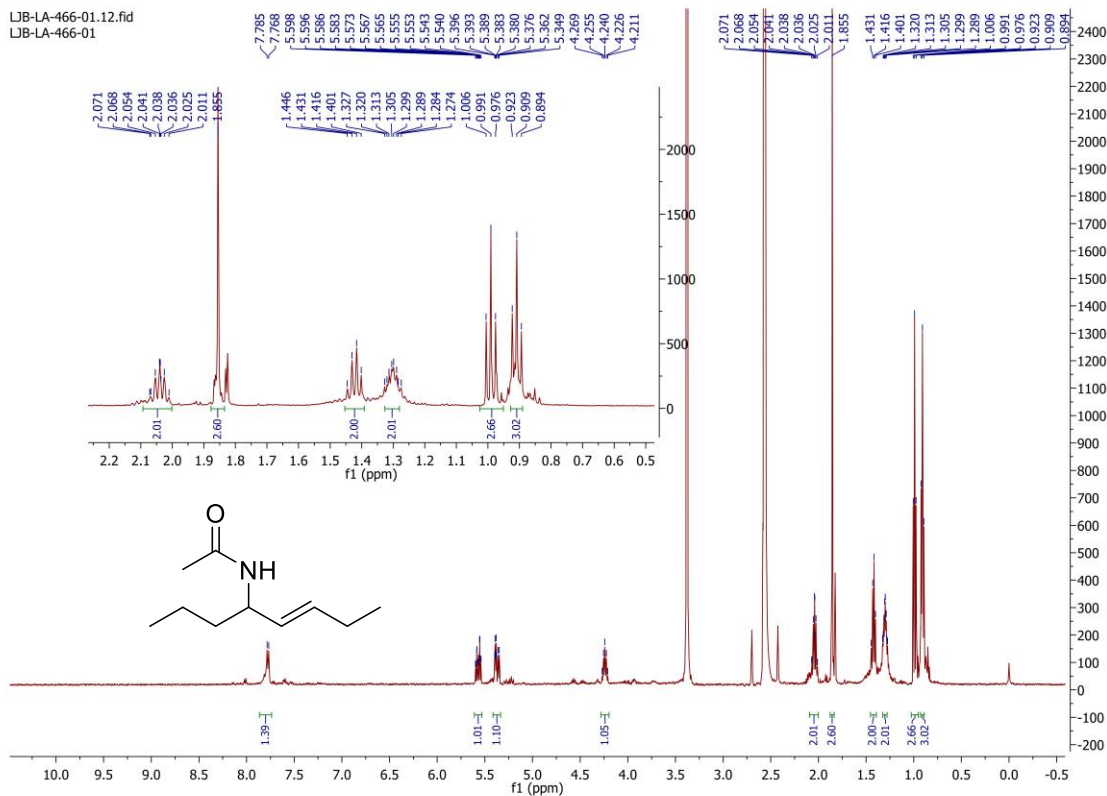
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HJB-LA-466-02
13C{1H} kurz
AV600a



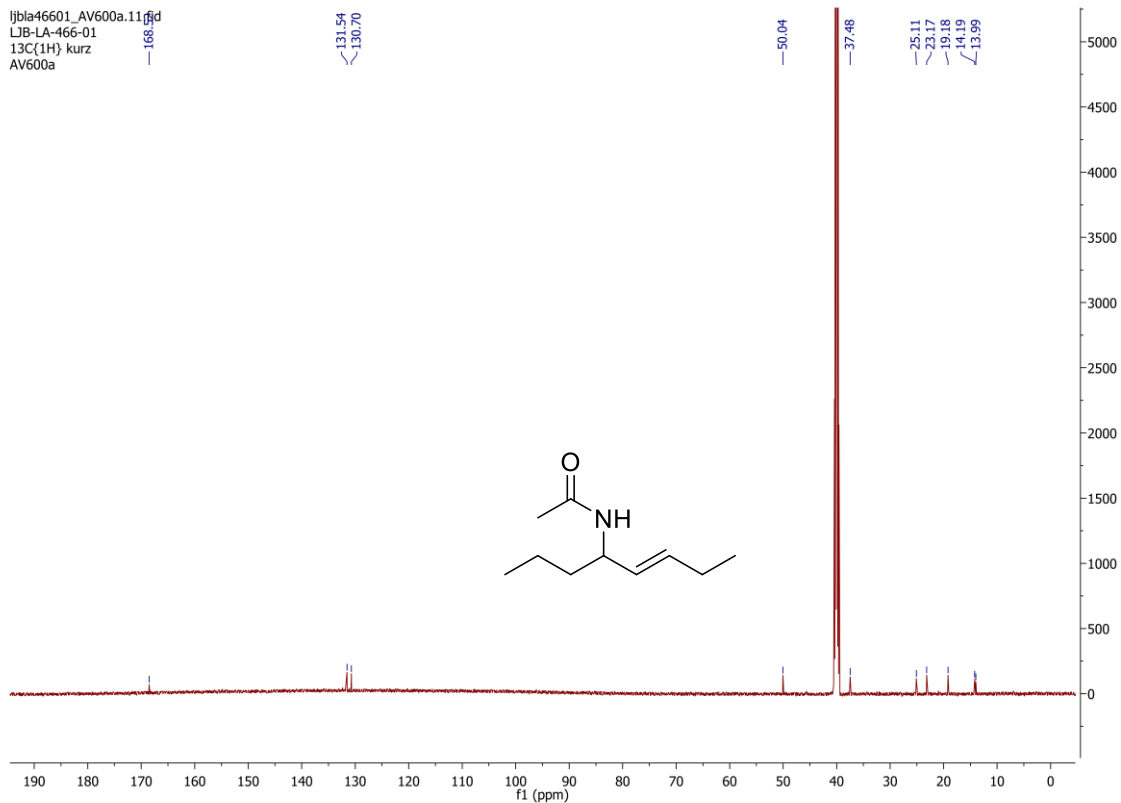
ljbla46602_AV600a.12.fid
HJB-LA-466-02
13C(1H)DEPT135
AV600a



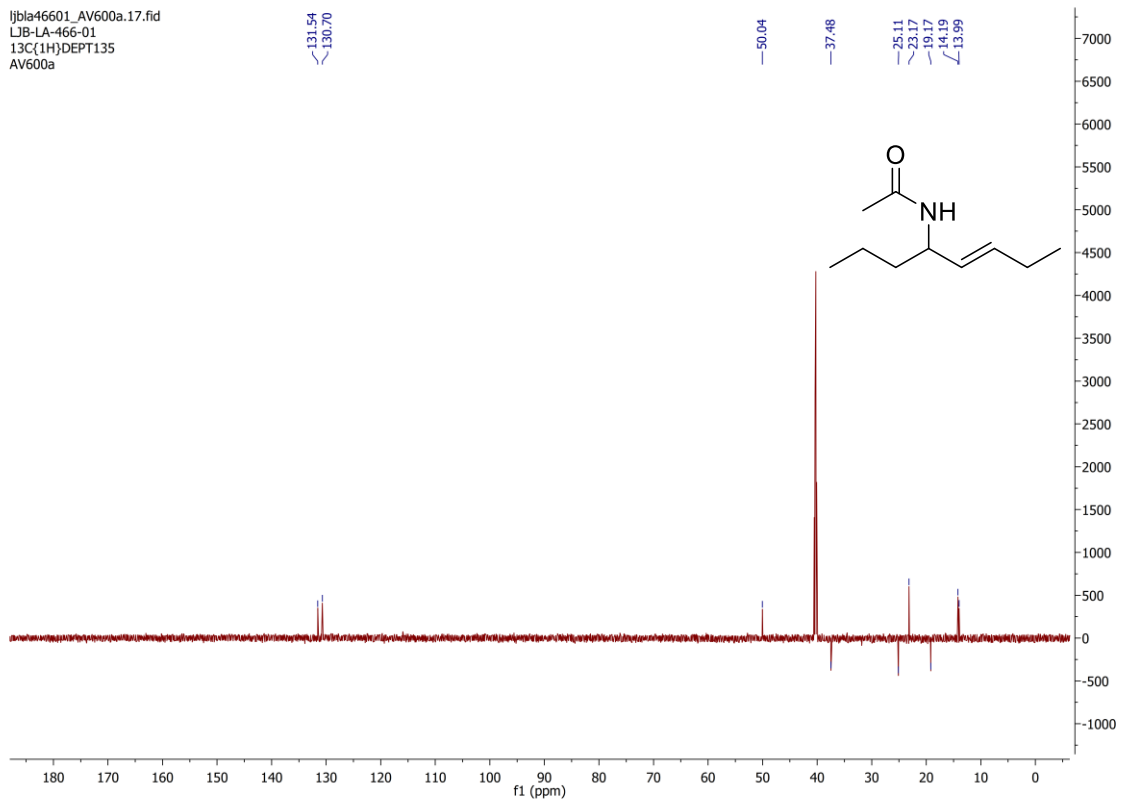
LJB-LA-466-01.12.fid
LJB-LA-466-01



ljbla46601_AV600a.11.fid
LJB-LA-466-01
13C{1H} kurz
AV600a



ljbla46601_AV600a.17.fid
LJB-LA-466-01
13C{1H}DEPT135
AV600a



| No. | MW. | Comment |
|-----|-----|---|
| 1 | 169 | Both structures seen below are possible Ref.-Spektr. Nr. U21741:146568b-00 LJB-LA-466-01 |
| 2 | 169 | Both structures seen below are possible Ref.-Spektr. Nr. U21742:146568b-00 LJB-LA-466-01 |
| 3 | 169 | Equal epak 1 |
| 4 | 169 | Equal peak 2 |

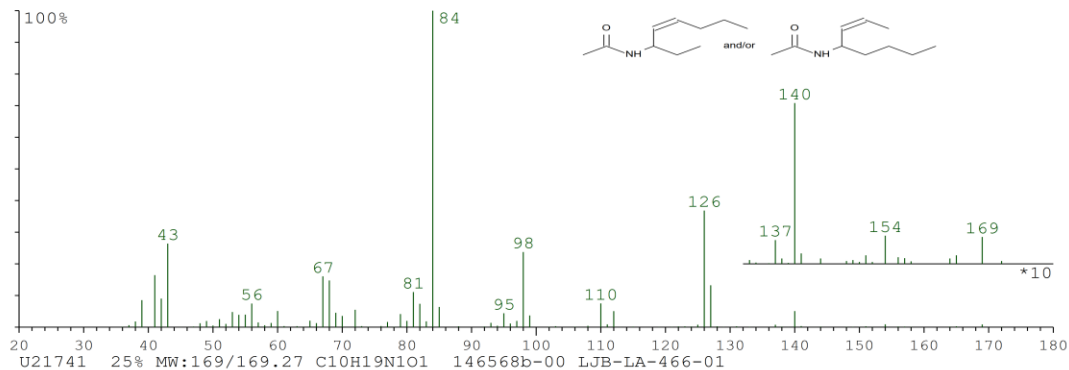
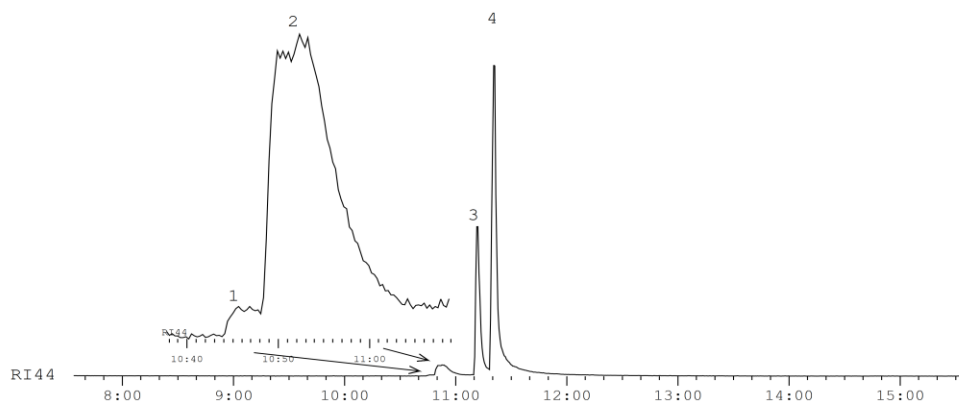
Other measurements will follow (HRMS)
Please call back if you can provide further information
Identification of isomers not possible via MS

9.04.2020
File: 146568b-00.raw

Analyse: LJB-LA-466-01
KMA: Liu, Sensheng

Messung: GC-MS
Ionisierung: GC-EI
Spektrometer: ISQ
Säule: MS 50 RTX-1
Länge: 30
Temp.: 35-10-285-5
GC-Nr.: -
MS-Nr.: 24585

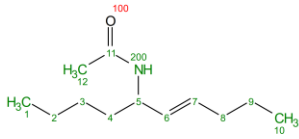
Auswerter: Margold (2242)



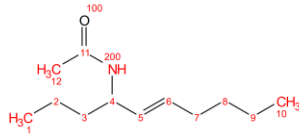
| | | | | | | | | | |
|----|-------|----|-------|-----|--------|-----|-------|-----|------|
| 36 | 0.14 | 59 | 1.32 | 83 | 1.83 | 119 | 0.08 | 148 | 0.09 |
| 37 | 0.62 | 60 | 5.10 | 84 | 100.00 | 121 | 0.10 | 149 | 0.12 |
| 38 | 1.79 | 61 | 0.33 | 85 | 6.38 | 122 | 0.24 | 150 | 0.06 |
| 39 | 8.53 | 63 | 0.31 | 88 | 0.28 | 123 | 0.25 | 151 | 0.27 |
| 41 | 16.43 | 64 | 0.17 | 92 | 0.21 | 124 | 0.21 | 152 | 0.06 |
| 42 | 9.02 | 65 | 2.04 | 93 | 1.36 | 125 | 0.76 | 154 | 0.89 |
| 43 | 26.40 | 66 | 1.27 | 94 | 0.44 | 126 | 36.83 | 156 | 0.21 |
| 45 | 0.03 | 67 | 16.05 | 95 | 4.39 | 127 | 13.23 | 157 | 0.18 |
| 46 | 0.05 | 68 | 14.75 | 96 | 1.15 | 128 | 0.26 | 158 | 0.08 |
| 47 | 0.20 | 69 | 4.54 | 97 | 1.97 | 129 | 0.08 | 164 | 0.17 |
| 48 | 1.23 | 70 | 3.54 | 98 | 23.73 | 130 | 0.06 | 165 | 0.27 |
| 49 | 1.94 | 72 | 5.49 | 99 | 3.69 | 131 | 0.27 | 169 | 0.85 |
| 50 | 0.50 | 73 | 0.32 | 102 | 0.11 | 133 | 0.12 | 172 | 0.09 |
| 51 | 2.52 | 74 | 0.14 | 103 | 0.30 | 134 | 0.04 | | |
| 52 | 1.02 | 76 | 0.34 | 106 | 0.14 | 136 | 0.02 | | |
| 53 | 4.78 | 77 | 1.63 | 108 | 0.41 | 137 | 0.75 | | |
| 54 | 3.90 | 78 | 0.11 | 110 | 7.50 | 138 | 0.17 | | |
| 55 | 3.95 | 79 | 4.14 | 111 | 0.87 | 139 | 0.03 | | |
| 56 | 7.46 | 80 | 1.98 | 112 | 5.06 | 140 | 5.08 | | |
| 57 | 1.49 | 81 | 11.09 | 113 | 0.05 | 141 | 0.33 | | |
| 58 | 0.59 | 82 | 7.38 | 115 | 0.16 | 144 | 0.17 | | |

U21741 25% MW:169/169.27 C10H19NO1 146568b-00 LJB-LA-466-01

main compound



minor compound



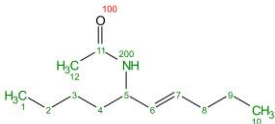
| | | | |
|------------------|--------------------------------|--|--|
| ELNA: | 4259 | | |
| Measured on: | 17.07.2020 | | |
| CHIFFRE: | LIB-LA-663-01 | | |
| Client: | Liu | | |
| Group: | Klößmann | | |
| Analyst: | Tobegen | | |
| Assignment Date: | 03.08.2020 | | |
| Amount: | 2 mg | | |
| Solvent: | DMSO | | |
| Reference: | solvent | | |
| Temperature: | 298K | | |
| Spectrometer: | Av600a | | |
| Experiments: | 1H, 13C[1H], HSQC, HMBC | | |
| | COSY, NOESY, 15N-HMBC, dept135 | | |

-This is a mixture of products. The signals are heavily overlapped in 1H, but thankfully many signals can be resolved in 13C.

-Therefore, the assignment is mainly based on predictions from the suggested products and due to some correlation peaks in 2D spectra.

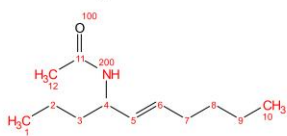
-Most important CH2-Group to assign the molecules is 2C from the smaller compound with the shift at ~19 ppm.

main compound



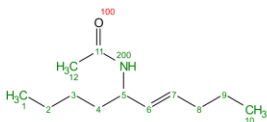
| Atom | δ (ppm) | Min..Max (ppm) | Predicted Shift | COSY | HSQC | HMBC | NOESY |
|-------|----------------|----------------|-----------------|-----------|---------|-----------------------|-------------------------|
| 1 C | 13.86 | 13.80..13.92 | 13.9 | | 1 | 2, 3', 3'', 4 | |
| H3 | 0.83 | 0.81..0.86 | 0.84 | 2 | 1 | 2, 3 | 2, 3', 3'' |
| 2 C | 21.48 | 21.42..21.54 | 23.2 | | 2 | 1, 4 | |
| H2 | 1.24 | 1.21..1.28 | 1.32, 1.34 | 1 | 2 | 1 | 1 |
| 3 C | 27.65 | 27.57..27.71 | 28.94 | | 3', 3'' | 1, 4, 5 | |
| H' | 1.21 | 1.16..1.26 | 1.25, 1.33 | | 3 | 1, 4, 5 | 1, 4, 5, 200 |
| H'' | 1.21 | 1.15..1.26 | 1.25, 1.33 | | 3 | 1, 4, 5 | 1, 4, 5, 200 |
| 4 C | 34.45 | 34.37..34.51 | 33.4 | | 4 | 3', 3'', 5, 6 | |
| H2 | 1.36 | 1.34..1.38 | 1.39, 1.57 | 5 | 4 | 1, 2, 3, 5, 6 | 3', 3'', 5, 6, 200 |
| 5 C | 49.87 | 49.84..49.90 | 53.19 | | 5 | 3', 3'', 4, 6, 7, 200 | |
| H | 4.15 | 4.10..4.19 | 4.18 | 4, 6, 200 | 5 | 3, 4, 6, 7, 11 | 3', 3'', 4, 6, 7, 200 |
| 6 C | 131.4 | 131.37..131.42 | 132.54 | | 6 | 4, 5, 7, 8 | |
| H | 5.3 | 5.26..5.33 | 5.27 | 5, 7 | 6 | 4, 5, 7, 8 | 4, 5, 8, 200 |
| 7 C | 129.3 | 129.27..129.32 | 132.43 | | 7 | 5, 6, 8, 9 | |
| H | 5.43 | 5.39..5.46 | 5.56 | 6, 8 | 7 | 5, 6, 8, 9 | 5, 8, 9, 200 |
| 8 C | 33.62 | 33.55..33.69 | 36.5 | | 8 | 6, 7, 9, 10 | |
| H2 | 1.93 | 1.89..1.97 | 2.08, 2.10 | 7, 9, 10 | 8 | 6, 7, 9, 10 | 6, 7, 10 |
| 9 C | 21.82 | 21.80..21.83 | 22.69 | | 9 | 7, 8, 10 | |
| H2 | 1.32 | 1.29..1.35 | 1.42 | 8, 10 | 9 | 7, 8, 10 | 7, 10 |
| 10 C | 13.38 | 13.32..13.44 | 13.63 | | 10 | 8, 9 | |
| H3 | 0.83 | 0.81..0.86 | 0.93 | 8, 9 | 10 | 8, 9 | 8, 9 |
| 11 C | 167.98 | 167.92..168.03 | 170.96 | | | 5, 12, 200 | |
| 12 C | 22.62 | 22.56..22.68 | 22.6 | | 12 | | |
| H3 | 1.78 | 1.76..1.79 | 2 | | 12 | 11 | 200 |
| 100 O | | | | | | | |
| 200 N | | | | | | | |
| H | 7.71 | 7.68..7.74 | 6.73 | 5 | | 5, 11 | 3', 3'', 4, 5, 6, 7, 12 |

minor compound

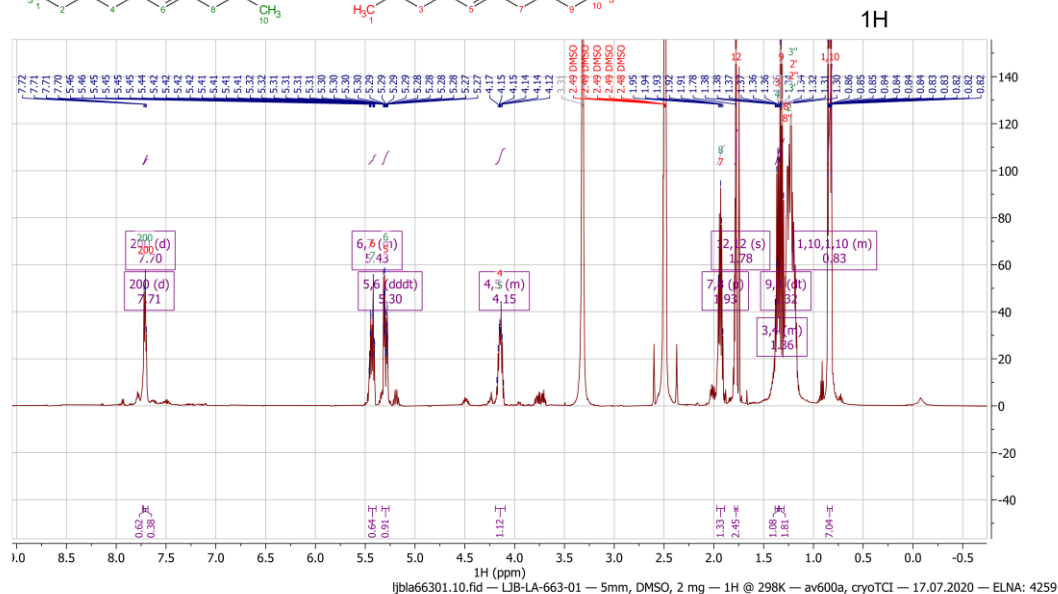
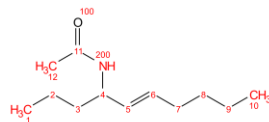


| Atom | δ (ppm) | Min..Max (ppm) | Predicted Shift | COSY | HSQC | HMBC | NOESY |
|------|----------------|----------------|-----------------|------------|---------|----------------|-------------------------|
| 1C | 13.64 | 13.58..13.70 | 14.02 | | 1 | 2', 2'', 3 | |
| H3 | 0.83 | 0.81..0.86 | 0.93 | 2', 2'' | 1 | 2, 3 | 2', 2'' |
| 2C | 18.61 | 18.53..18.67 | 19.63 | | 2', 2'' | 1, 3, 4 | |
| H' | 1.2 | 1.17..1.23 | 1.26, 1.38 | 1 | 2 | 1 | 1, 3, 4, 200 |
| H'' | 1.2 | 1.17..1.23 | 1.26, 1.38 | 1 | 2 | 1 | 1, 3, 4, 200 |
| 3C | 36.92 | 36.83..36.99 | 34.71 | | 3 | 1, 4, 5 | |
| H2 | 1.36 | 1.34..1.38 | 1.39, 1.57 | 4 | 3 | 1, 2, 4, 5 | 2', 2'', 4, 5, 200 |
| 4C | 49.56 | 49.54..49.59 | 53.3 | | 4 | 3, 5, 6, 200 | |
| H | 4.15 | 4.10..4.19 | 4.18 | 3, 5, 200 | 4 | 2, 3, 5, 6, 11 | 2', 2'', 3, 5, 6, 200 |
| 5C | 131.17 | 131.15..131.20 | 132.57 | | 5 | 3, 4, 6, 7 | |
| H | 5.3 | 5.26..5.33 | 5.27 | 4, 6 | 5 | 3, 4, 6, 7 | 3, 4, 7, 200 |
| 6C | 129.46 | 129.43..129.48 | 131.33 | | 6 | 4, 5, 7, 9 | |
| H | 5.43 | 5.39..5.46 | 5.56 | 5, 7 | 6 | 4, 5, 7, 8, 9 | 4, 7, 8', 8'', 9, 200 |
| 7C | 31.18 | 31.09..31.26 | 33.66 | | 7 | 5, 6, 8', 8'' | |
| H2 | 1.93 | 1.89..1.97 | 1.99, 2.01 | 6, 8', 8'' | 7 | 5, 6, 8, 9, 10 | 5, 6, 8', 8'', 9, 10 |
| 8C | 30.85 | 30.72..30.92 | 30.73 | | 8', 8'' | 6, 7, 10 | |
| H' | 1.26 | 1.24..1.29 | 1.36 | 7 | 8 | 7, 9 | 6, 7, 10 |
| H'' | 1.26 | 1.24..1.28 | 1.36 | 7 | 8 | 7, 9 | 6, 7, 10 |
| 9C | 21.84 | 21.78..21.90 | 22.2 | | 9 | 6, 7, 8', 8'' | |
| H2 | 1.32 | 1.29..1.35 | 1.38 | 10 | 9 | 6 | 6, 7, 10 |
| 10C | 13.7 | 13.64..13.76 | 13.96 | | 10 | 7 | |
| H3 | 0.83 | 0.81..0.86 | 0.89 | 9 | 10 | 8 | 7, 8', 8'', 9 |
| 11C | 167.98 | 167.92..168.03 | 170.96 | | | 4, 12, 200 | |
| 12C | 22.62 | 22.56..22.68 | 22.6 | | 12 | | |
| H3 | 1.78 | 1.76..1.79 | 2 | | 12 | 11 | 200 |
| 100O | | | | | | | |
| 200N | | | | | | | |
| H | 7.7 | 7.68..7.73 | 6.73 | 4 | | 4, 11 | 2', 2'', 3, 4, 5, 6, 12 |

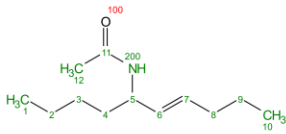
main compound



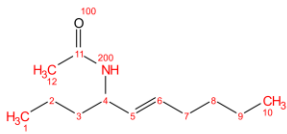
minor compound



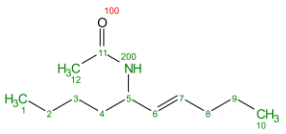
main compound



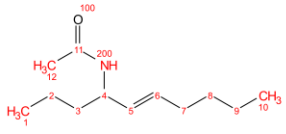
minor compound



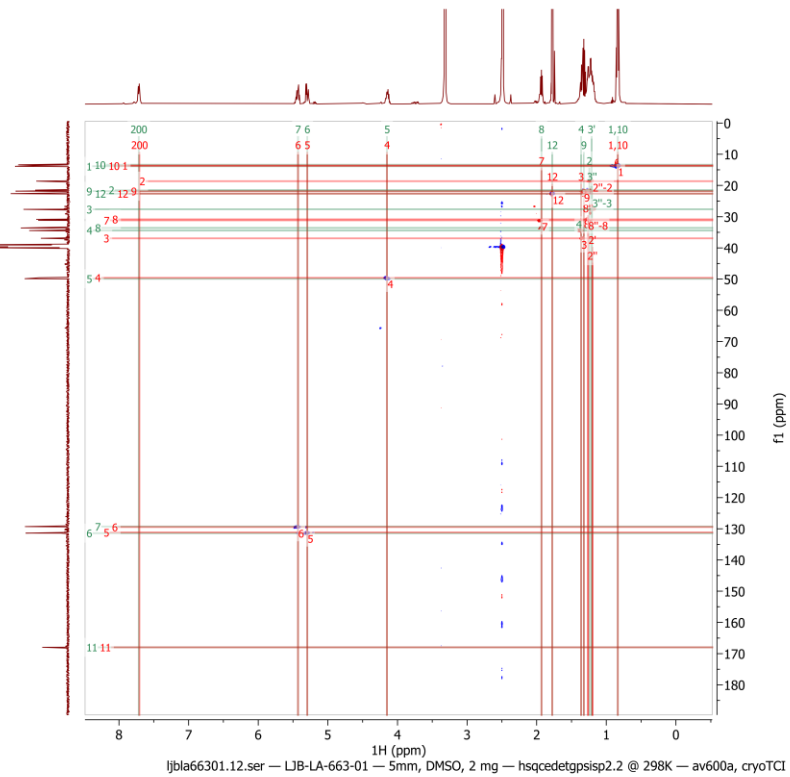
main compound



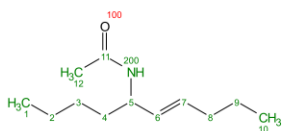
minor compound



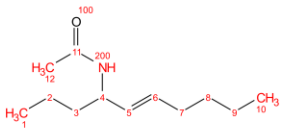
HSQC (hsqcedetgpsisp2.4)



main compound

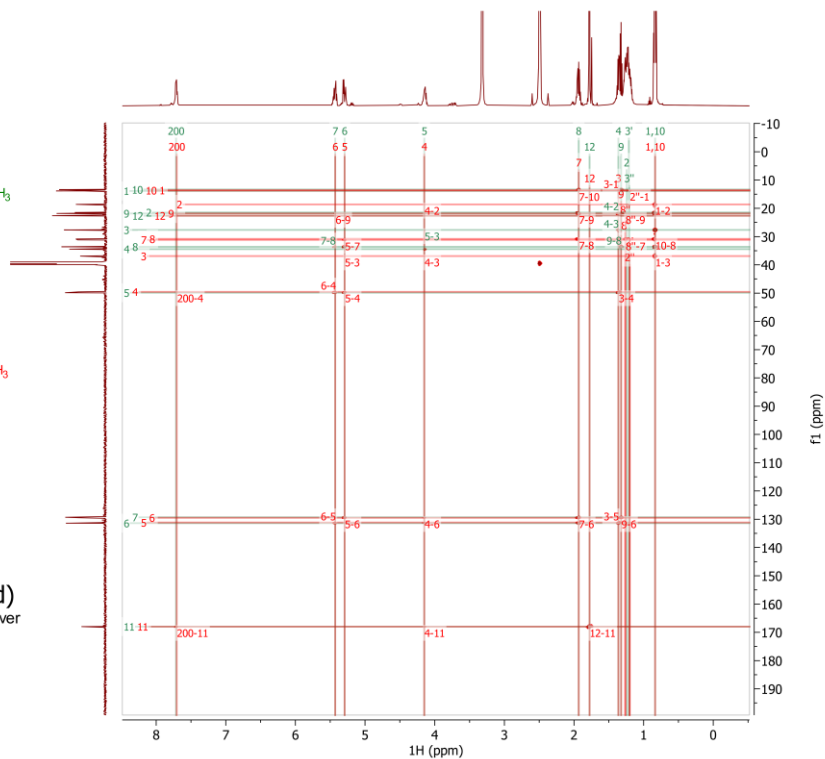


minor compound

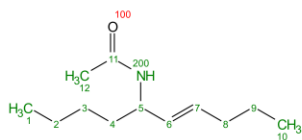


HMBC (hmbcetgpl3nd)

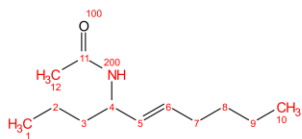
See HMBC-arrows with mouse over atoms.



main compound

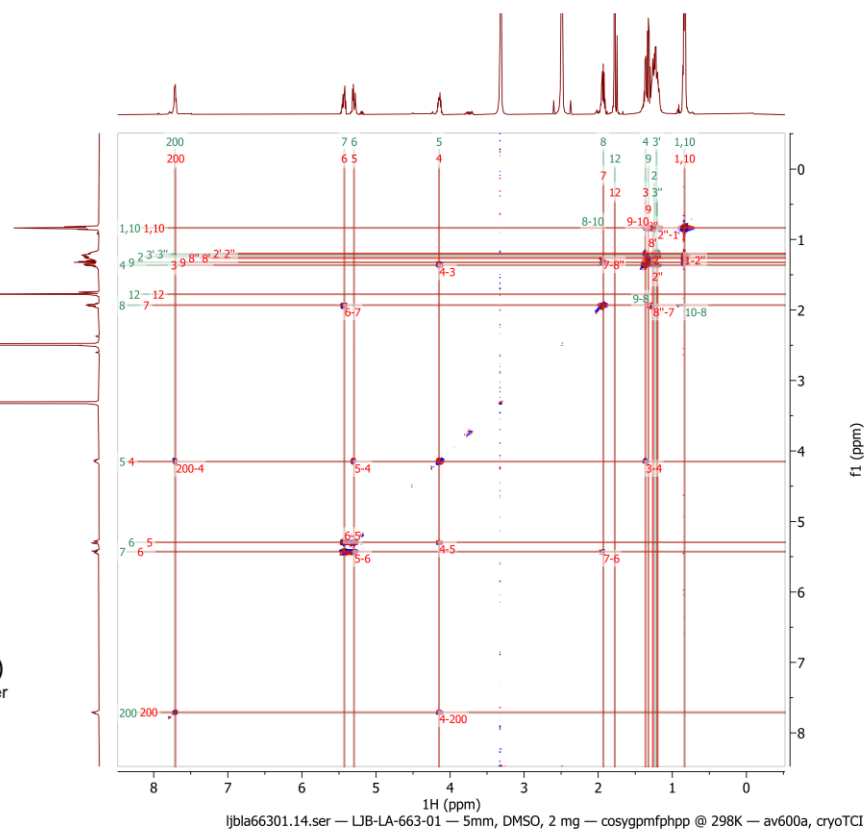


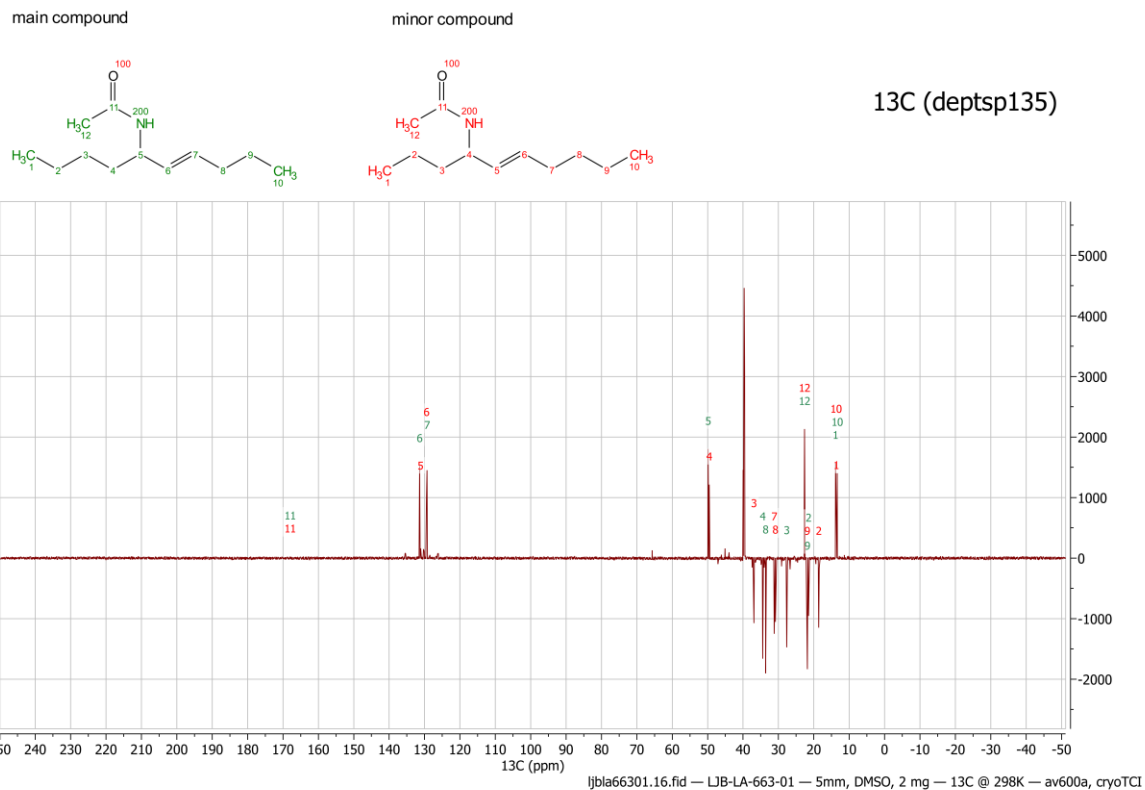
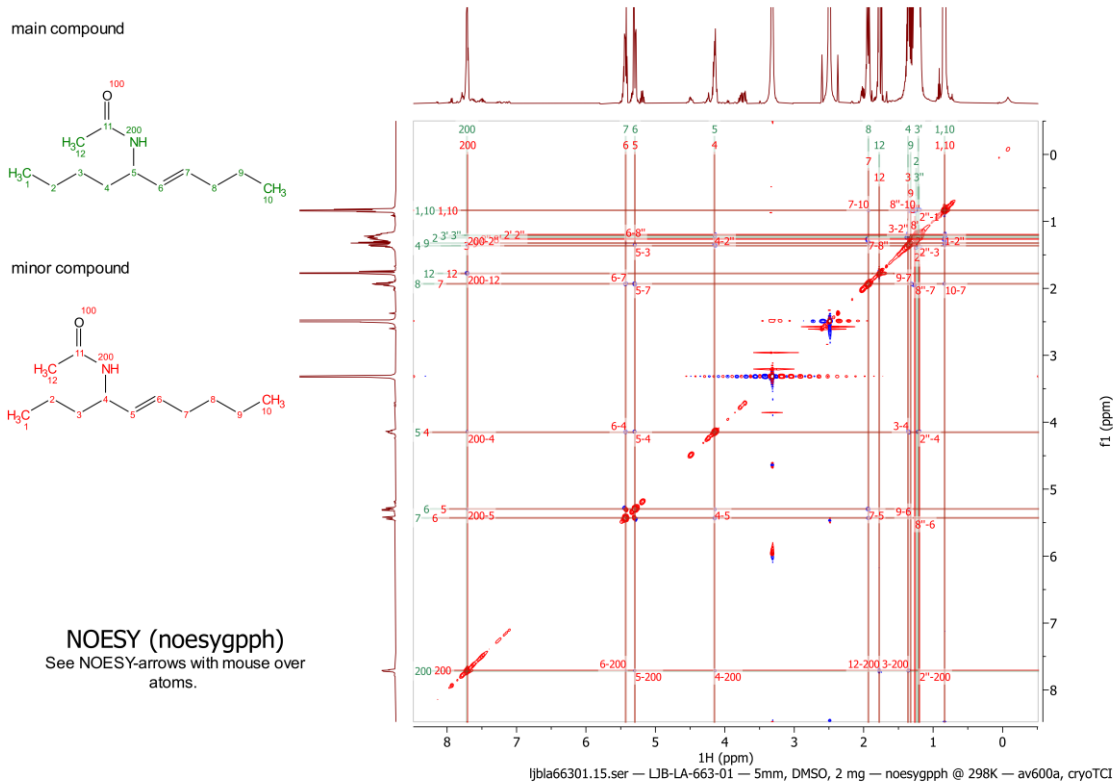
minor compound



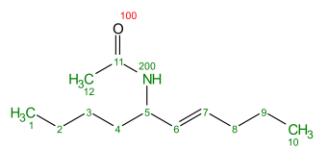
COSY (cosygmfpfpp)

See COSY-arrows with mouse over atoms.

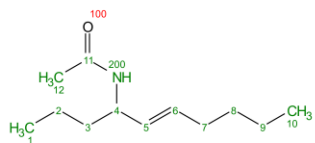




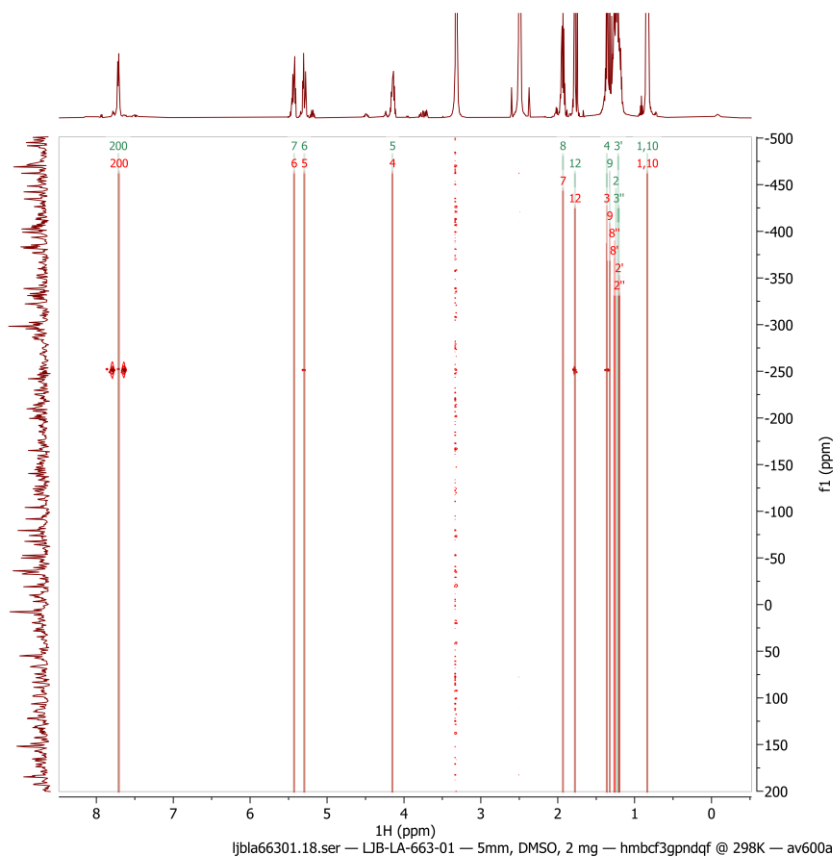
main compound



minor compound



15N-HMBC (hmbcf3gpndqf)



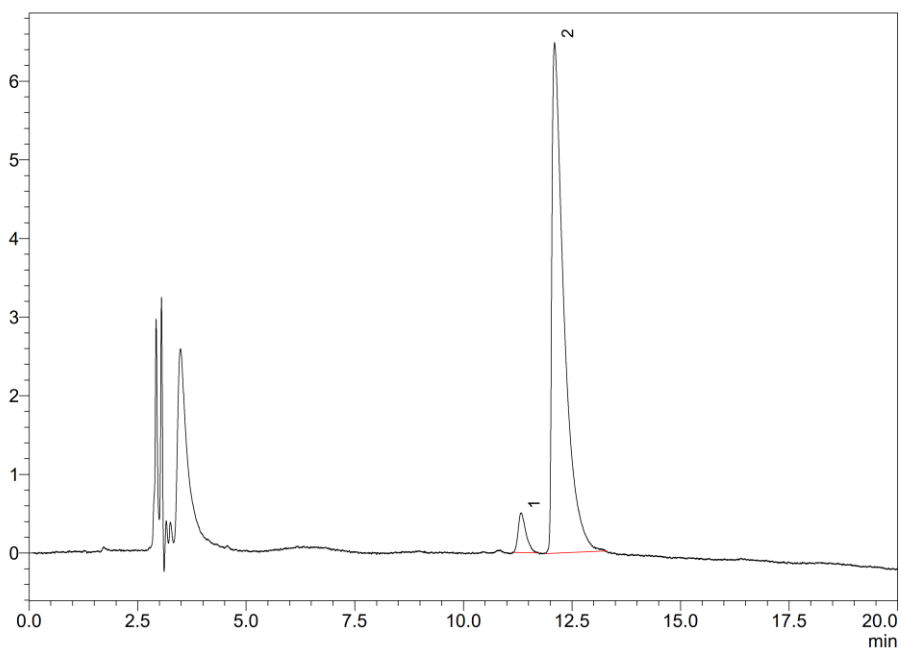
Gerät : NP-3

Operator : Ke
Sample Name : LJB-LA-663-01-10 gesamt
Vial # : 44
Injection Volume : 5 uL
Data File Name : LJB-LA-663-01-G10-03.lcd
Method File Name : Liu.lcm

Data Acquired: 8/19/2020 2:56:52 PM

5 µL LJB-LA-663-01-10, nach Aufarbeitung
20 µL in 180 µL MTBE
250 mm Multo Krom Si 3 µm, 4.6 mm i.D
2705-18
n-Heptan/ MTBE = 30:70
1.0 mL/min, 13.8 MPa, 308 K
UV, 210 nm

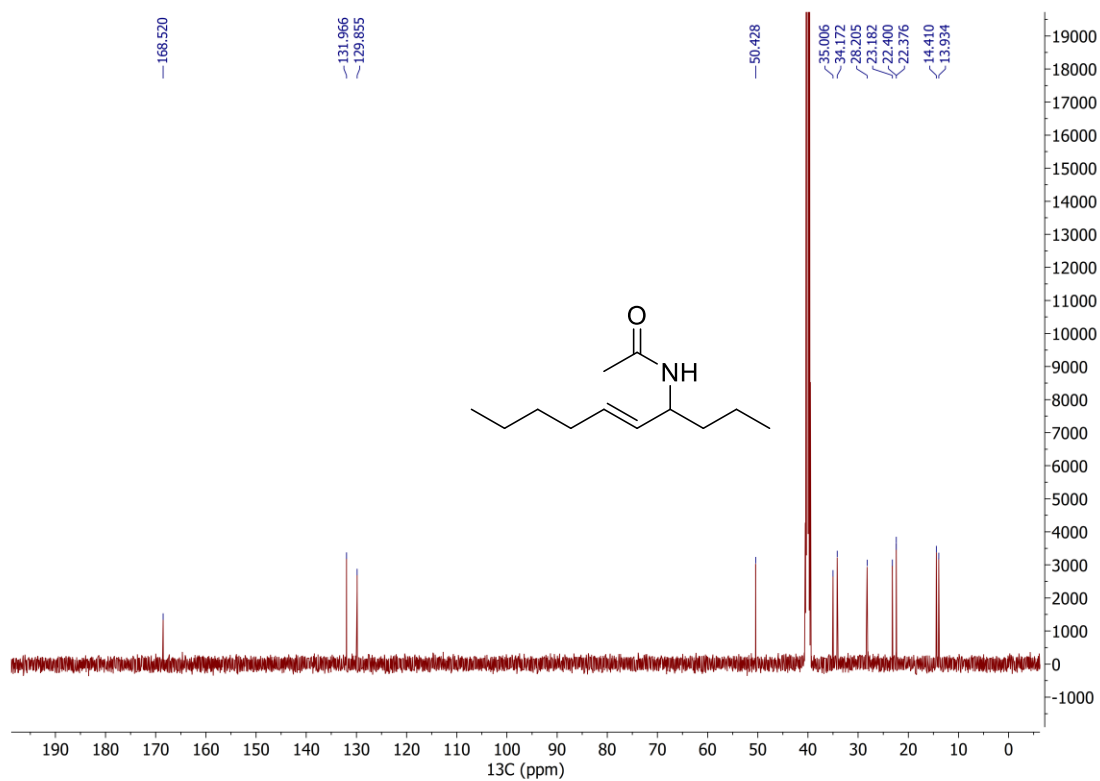
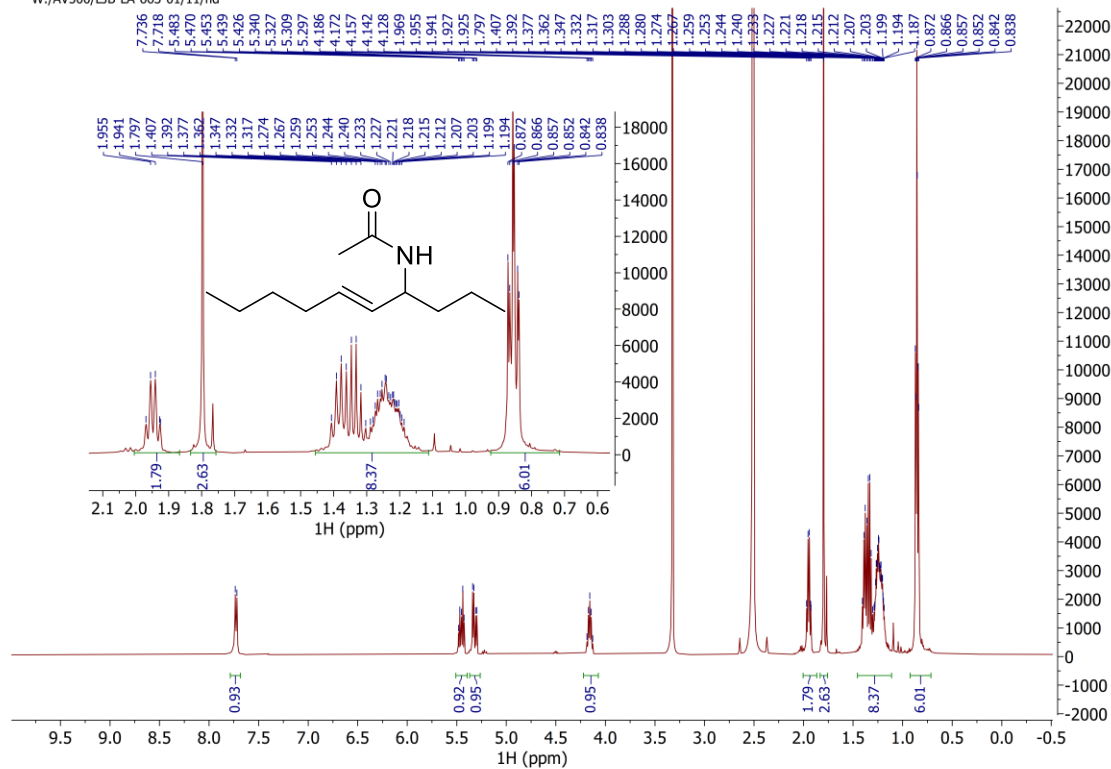
mAU

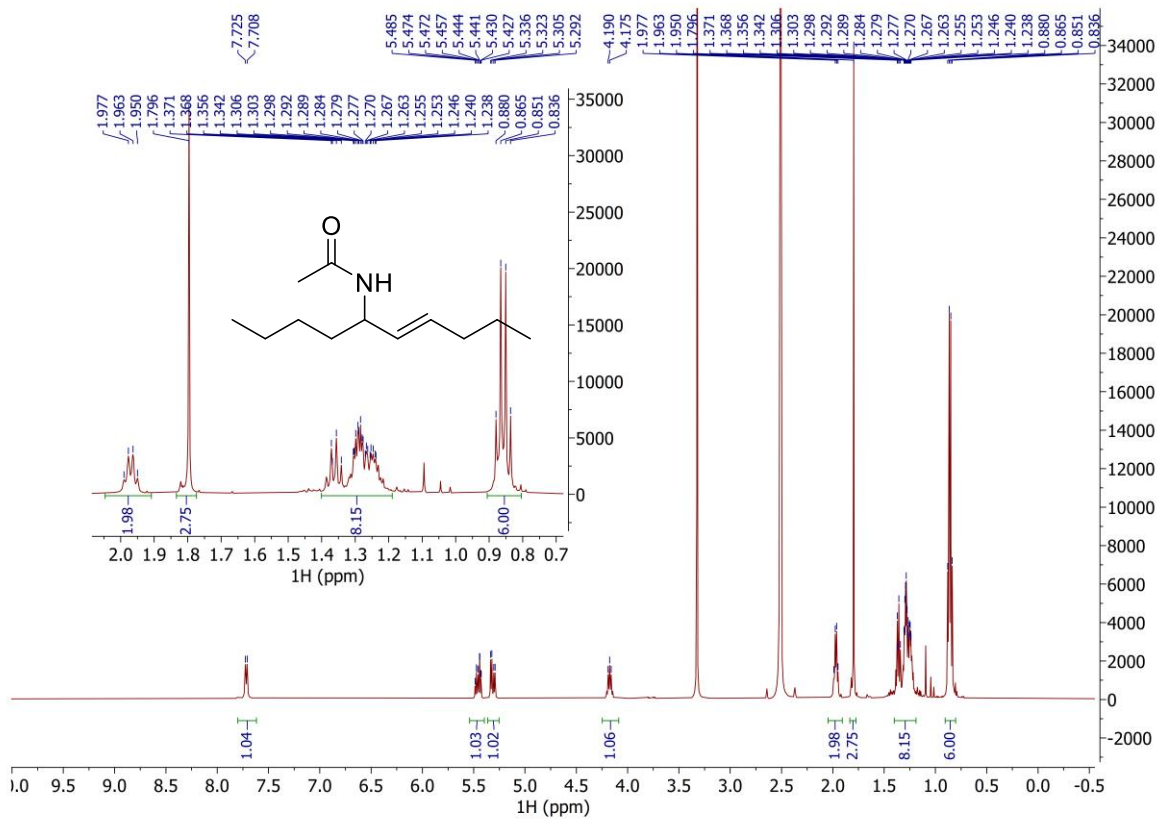
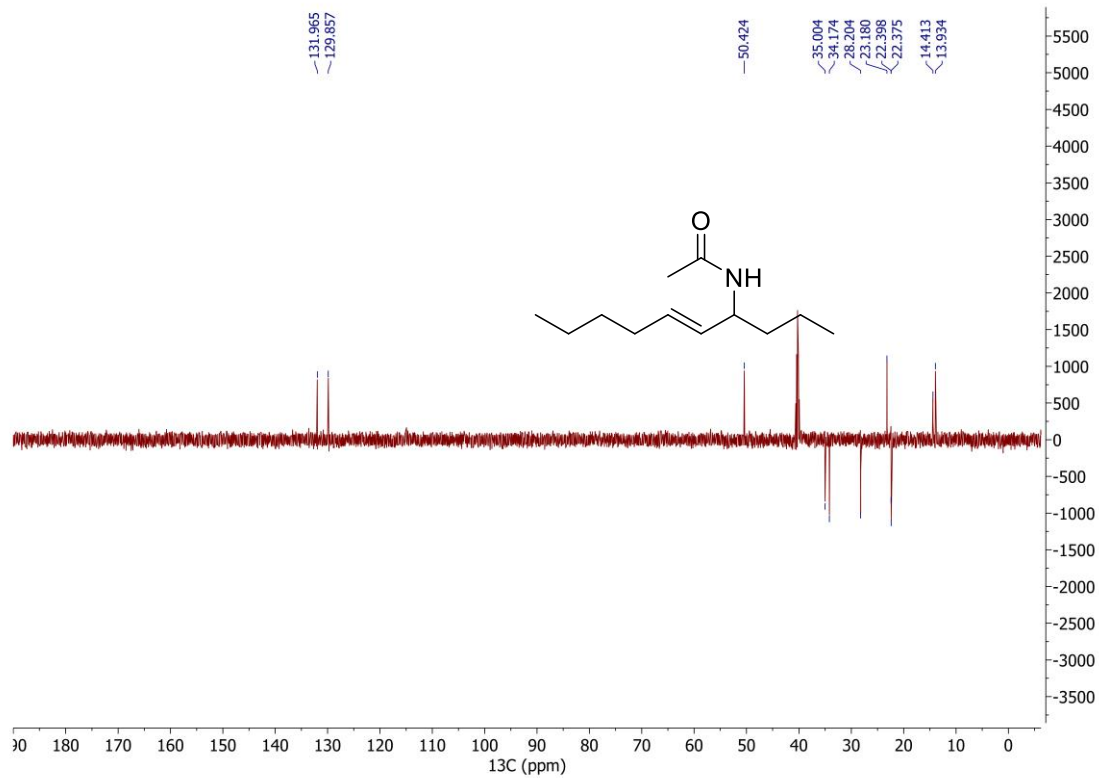


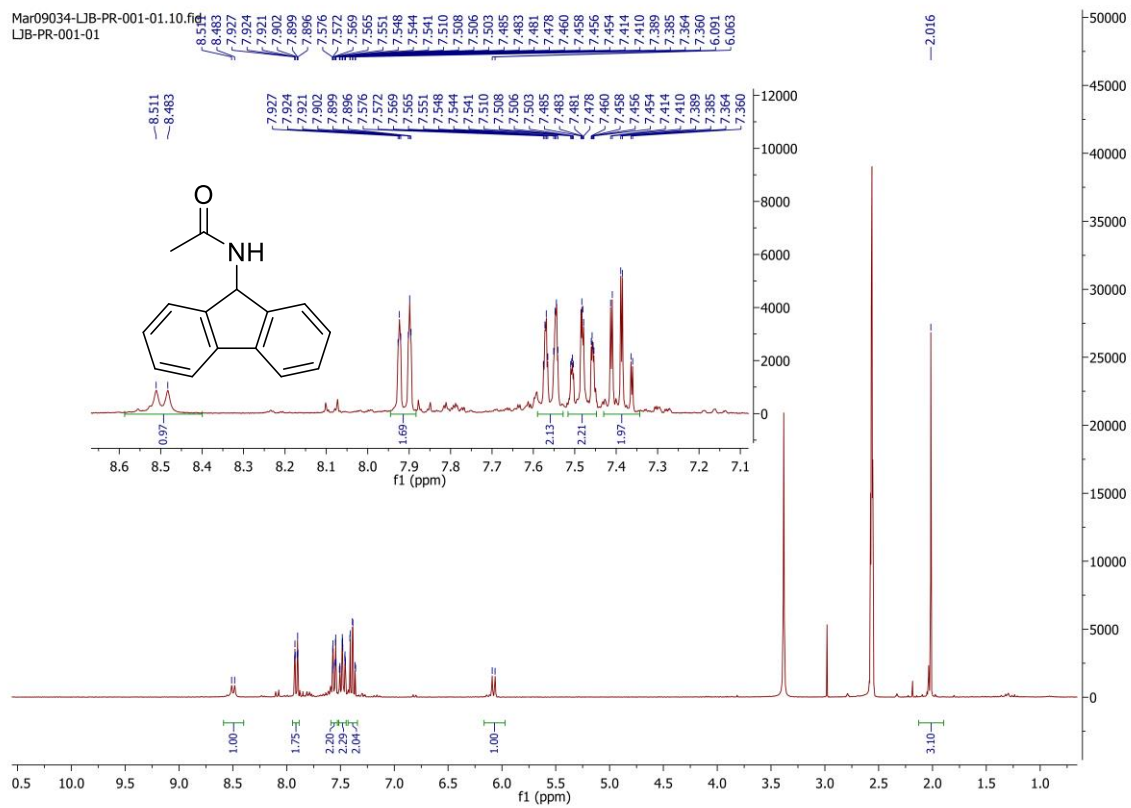
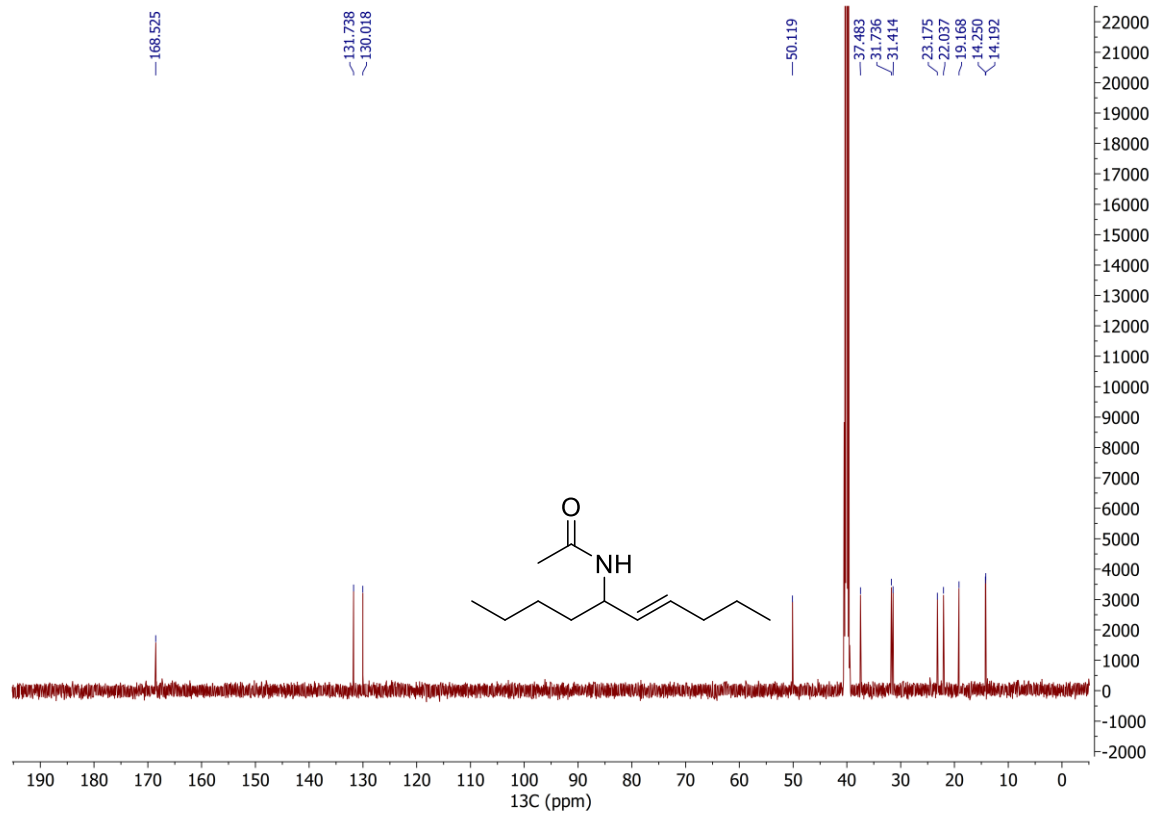
1 210nm,4nm

| PDA Ch1 210nm | | | |
|---------------|-----------|--------|-----------|
| Peak # | Ret. Time | Area % | Name |
| 1 | 11.33 | 4.72 | |
| 2 | 12.10 | 95.28 | 1. isomer |
| Total | | 100.00 | |

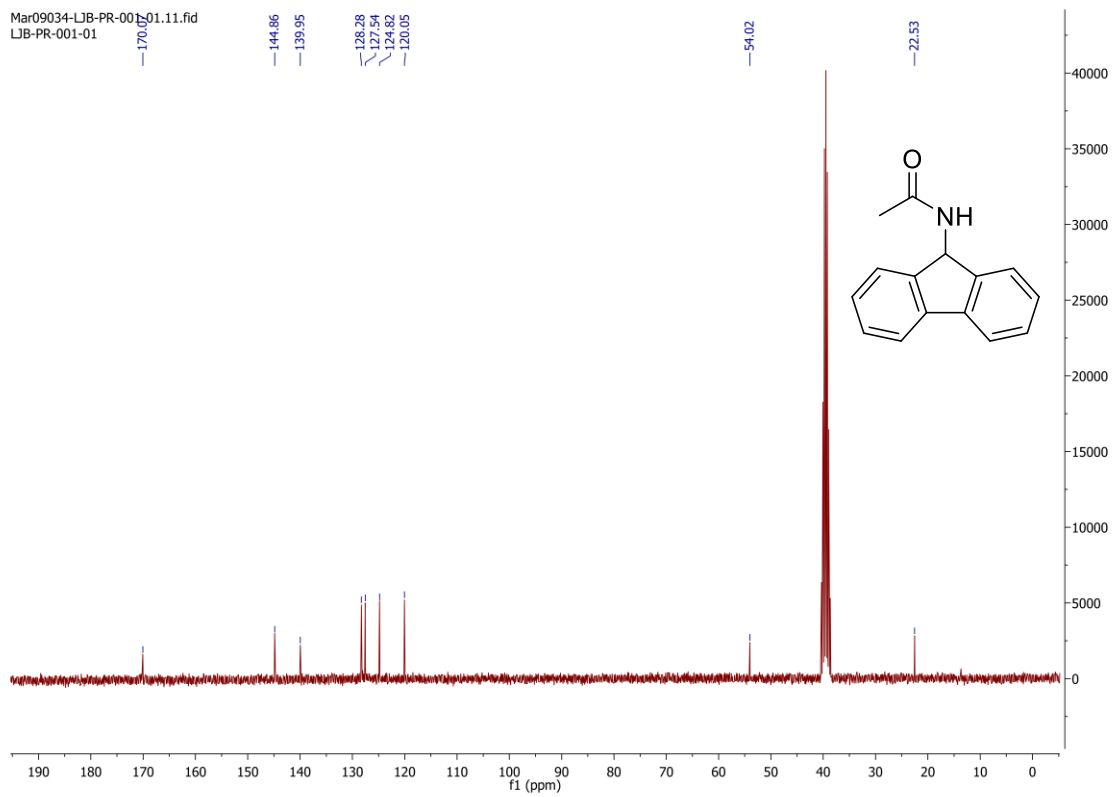
W:/AV500/LJB-LA-663-01/11/fid







Mar09034-LJB-PR-001.11.fid
LJB-PR-001-01



Reference

- [1] G. R. Fulmer, A. J. M. Miller, N. H. Sherden, H. E. Gottlieb, A. Nudelman, B. M. Stoltz, J. E. Bercaw, K. I. Goldberg, *Organometallics* 2010, 29, 2176.
- [2] T. A. Schaub, T. Mekelburg, P. O. Dral, M. Miehlich, F. Hampel, K. Meyer, M. Kivala, *Chem. Eur. J.* 2020, 26, 3264-3269.